Tim Drexler, 5/02/13

#4238

Quality Assurance Project Plan

ADDENDUM

AECOM
SUPERVISING CONTRACTOR

FOR:
HAMILTON SUNDSTRAND
AREA 9/10 SOUTHEAST ROCKFORD GROUNDWATER CONTAMINATION SUPERFUND SITE
ROCKFORD, ILLINOIS

April 2013

QUALITY ASSURANCE PROJECT PLAN ADDENDUM

SE ROCKFORD AREA 9/10
WINNEBAGO COUNTY, ILLINOIS

Prepared by: AECOM

_____ _____
Mr. Scott Moyer, P.G.
UTAS Project Coordinator Date

_____ _____
Mr. Peter Hollatz, P.E.
AECOM Project Manager Date

_____ _____
Mr. Gregory A. Malzone
AECOM QA Manager/Project Chemist Date

_____ _____
Mr. Timothy Drexler
U.S. EPA Region 5 Remedial Project Manager - CERCLA Date

_____ _____
Mr. Richard Byvik
U.S. EPA Region 5 Quality Assurance Reviewer Date

_____ _____
Mr. Doyle Wilson
IEPA Project Coordinator Date

_____ _____
Ms. Marie Meidhof
Accutest Project Manager Date

_____ _____
Ms. Melanie Levesque
Eurofins AirToxics, Inc. QA Manager Date

1.0 PROJECT BACKGROUND

AECOM Technical Services, Inc. (AECOM) was authorized by UTC Aerospace Systems (UTAS, fka Hamilton Sundstrand or HS) to prepare an Addendum to the Quality Assurance Project Plan (QAPP) associated with the Remedial Action (RA) being performed at the Area 9/10 portion of the Southeast Rockford Groundwater Superfund site, Rockford, Illinois (Site). This document serves to revise/update the approved QAPP submitted in 2008 by Stantec Consulting Services, Inc. as part of the Remedial Design Work Plan for the RA. The revised and updated information provided in this Addendum is required to address recent changes in project personnel and organizations.

AECOM has recently been contracted by UTAS to serve as its remediation contractor to perform activities required in the Operation, Maintenance and Monitoring Plan (OM&M Plan, Stantec 2007), and other RA activities as they are deemed necessary by UTAS. In addition, a new analytical laboratory has been selected for the project. These changes have resulted in changes to the project organizational chart. Aside from the organizational changes, this Addendum modifies the QAPP to reflect the statements in the EPA letter of December 5, 2011 indicating that the EPA RCRA program has deferred the remaining contaminated groundwater issue to the EPA CERCLA program in an effort to avoid duplicative programmatic review during implementation of the remedies.

UTAS entered into a Consent Decree (CD) with the United States Environmental Protection Agency (USEPA) and Illinois Environmental Protection Agency (IEPA) on September 2, 1998 for the completion of a RA for source control at the UTAS property within Area 9/10. UTAS is in the process of performing certain actions in a manner consistent with the Record of Decision (OU3 ROD 2002) and the performance standards and requirements of the CD as more fully described in the controlling documents, including: the Statement of Work (SOW 2008), Final 100% Remedial Design (Stantec, 2007), Remedial Action Work Plan (RAWP, Stantec 2008) and EPA-approved Remedial Action Process Flow Diagram (RAPFD).

Currently operating along the southeast boundary of the GMZ is the Phase 1 AS/SVE system. The Phase 1 AS/SVE system commenced full-scale operation on December 7, 2009. The Phase 2 AS/SVE system, currently operating within identified groundwater source areas, commenced full scale operation in March 2011.

2.0 PROJECT ORGANIZATION

This section provides the project organization and associated roles including key personnel, and descriptions of duties. Figure 2 provides an organizational chart depicting the overall management approach to these activities.

2.1 USEPA

The USEPA Remedial Project Manager (RPM) is the primary point of contact for the USEPA. This individual has control over the administrative and technical aspects of the overall RA effort on behalf of the USEPA, including those regarding CERCLA matters. The USEPA RPM will

coordinate activities with the IEPA Project Manager (PM) and UTAS. The USEPA designated RPM is as follows:

Mr. Timothy Drexler
United States Environmental Protection Agency, Region 5
77 West Jackson Blvd.
Chicago, Illinois 60604
T(312) 353-4367 F(312) 353-5541 or F(312) 886-4071
E-mail: Drexler.Timothy@epa.gov

2.2 IEPA

The IEPA will provide support to the USEPA and UTAS during the performance of the RA effort. The IEPA will work closely with the USEPA as a co-signatory on the CD prescribing the RA effort. The IEPA will provide technical and administrative oversight of the RA in conjunction with the USEPA. The IEPA has identified its project manager to be as follows:

Mr. Doyle W. Wilson
National Priorities List Unit
Federal Sites Remediation Section
Division of Remediation Management
Bureau of Land
Illinois Environmental Protection Agency
1021 North Grand Avenue East
P.O. Box 19276
Springfield, Illinois 62794-9276
T 217-782-7592 F(815) 223-1344
E-mail: Doyle.Wilson@illinois.gov

Mr. Wilson will be supported by other IEPA personnel as necessary.

2.3 UTAS Project Manager

The UTAS Project Coordinator is the designated individual to interact with USEPA and IEPA with regard to the RA efforts. The UTAS Project Coordinator will be supported by additional UTAS personnel as well as primary and secondary subcontracted entities. The combination of these entities under the direction of the UTAS Project Coordinator will be responsible for the implementation of the activities identified in the CD. The UTAS Project Coordinator for the Site is as follows:

Mr. Scott Moyer, P.G.
UTAS
10501 Ray Drive
Roscoe, Illinois 61073
T(815) 270-0660 F(860) 660-5764
E-mail: Scott.Moyer@utc.com

2.4 AECOM Project Manager

The UTAS Project Coordinator will be supported by personnel from AECOM, UTAS's primary contractor, for the RA activities. AECOM has designated the following individual as the Project Manager (PM) responsible for the RA:

Mr. Peter Hollatz, PE
AECOM
27755 Diehl Rd., Suite 100
Warrenville, IL 60555
T 630-836-1700 F 630-836-1711
E-mail: Peter.Hollatz@aecom.com

Mr. Hollatz will be supported by other AECOM technical, administrative, quality, and health and safety staff. In addition to AECOM personnel, various subcontractors and suppliers will be utilized in the performance of the RA effort.

2.5 Laboratory Project Managers

Accutest, Dayton, New Jersey will provide analytical laboratory services for the soil and groundwater analysis. The laboratory project manager for the efforts will be:

Marie Meidhof
Accutest Laboratories
2235 Route 130
Dayton, NJ 08810
T: 732.355.4552 | F: 732.329.3499
mariem@accutest.com

Eurofins Air Toxics, Ltd., of Folsom, California, will provide analytical laboratory services for the air analyses. The laboratory project manager for the RA efforts will be:

Ms. Melanie Levesque
Eurofins Air Toxics, Ltd.
180-B Blue Ravine Rd
Folsom, CA 95630
T (916) 985-1000 F (916) 985-1020
M.Levesque@airtoxics.com

2.6 AECOM Quality Assurance Manager

The Quality Assurance (QA) Manager will be responsible for ensuring that data collection and analysis is conducted in a representative manner. As appropriate, problems identified by the QA Manager will be resolved in consultation with the appropriate laboratory representative. The QA Manager will also provide third-party data validation services for this project.

Mr. Gregory A. Malzone

AECOM
Gulf Tower
707 Grant Street
5th Floor
Pittsburgh, PA 15219
D 412.316.3524 F 412.297.5000
E-mail: greg.malzone@aecom.com

2.7 Distribution List

The following individuals will receive copies of the approved QAPP and subsequent revisions:

Mr. Timothy Drexler
Remedial Project Manager, Superfund Division
United States Environmental Protection Agency,
Region 5
77 West Jackson Blvd
Chicago, IL 60604

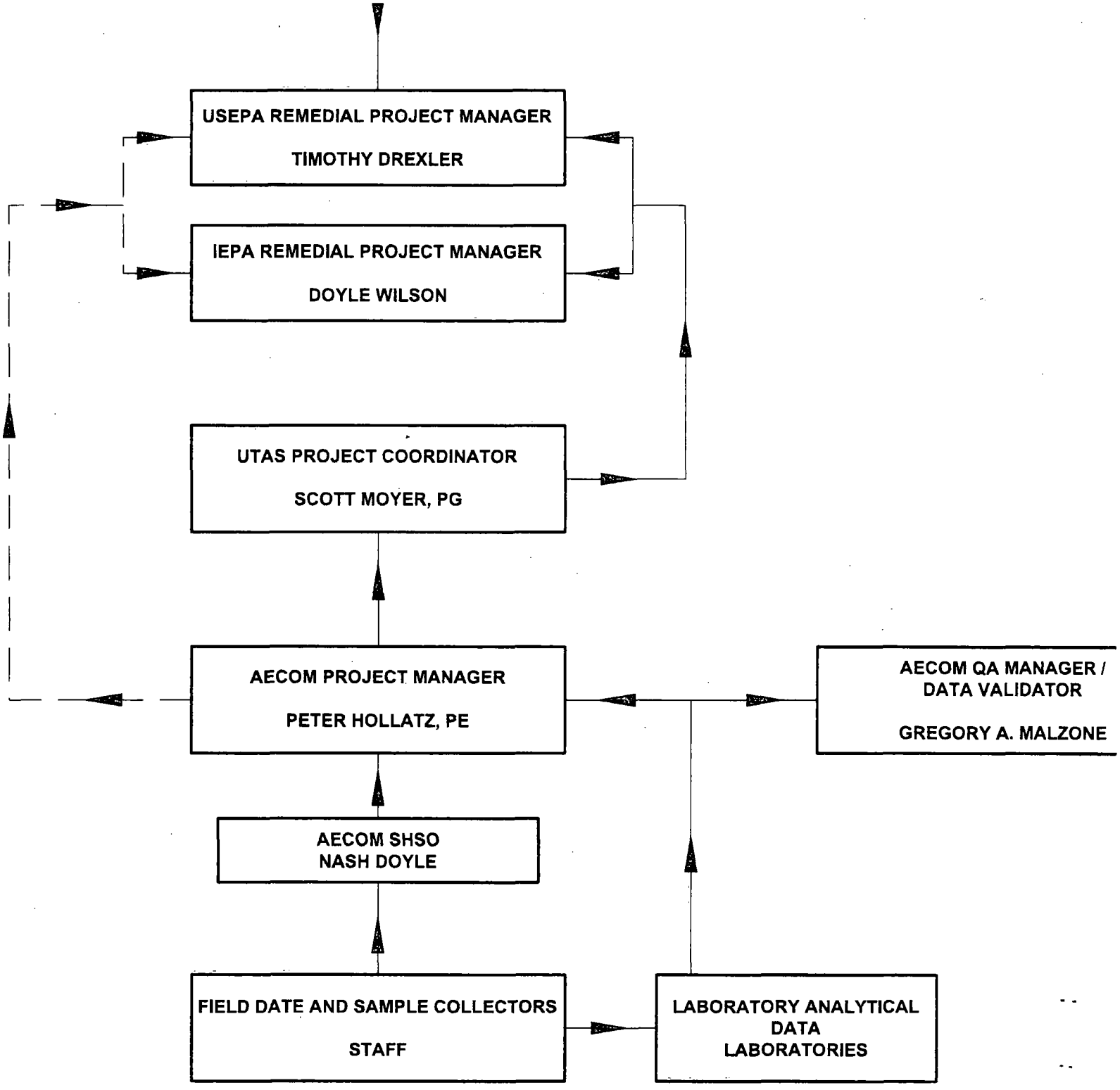
Mr. Scott Moyer, P.G.
Project Coordinator
United Technologies Corporation/Hamilton Sundstrand
10501 Ray Drive
Roscoe, Illinois 61073

Ms. Victoria Haines
Assistant General Counsel
Hamilton Sundstrand
4747 Harrison Avenue
P.O. Box 7002
Rockford, Illinois 61125-7002

Mr. Doyle W. Wilson
National Priorities List Unit
Federal Sites Remediation Section
Division of Remediation Management
Bureau of Land
Illinois Environmental Protection Agency
1021 North Grand Avenue East
P.O. Box 19276
Springfield, Illinois 62794-9276

Mr. Peter Hollatz, PE
Engineer
AECOM
27755 Diehl Road, Suite 100
Warrenville, IL 60555

FIGURE 2
REVISED ORGANIZATIONAL CHART


USEPA REMEDIAL PROJECT MANAGER
TIMOTHY DREXLER
IEPA REMEDIAL PROJECT MANAGER
DOYLE WILSON
UTAS PROJECT COORDINATOR
SCOTT MOYER, PG
AECOM PROJECT MANAGER
PETER HOLLATZ, PE
AECOM QA MANAGER / DATA VALIDATOR
GREGORY A. MALZONE
AECOM SHSO
NASH DOYLE
FIELD DATE AND SAMPLE COLLECTORS
STAFF
LABORATORY ANALYTICAL DATA LABORATORIES

APPENDIX A
Accutest Sample Label and Chain of Custody Form

Client Proj: By
Sample ID:
Date: Time:
Analysis:
Preservative:

Custody Seal
333

Custody Seal
334

CHAIN OF CUSTODY

PAGE ___ OF ___

ACCUTEST Laboratories
2235 Route 130, Dayton, NJ 08810
TEL: 732-329-0200 FAX: 732-329-3499/3480
www.accutest.com

FED-EX Tracking # | Bottle Order Control #

Accutest Quote # | Accutest Job #

Client / Reporting Information

Company Name

Street Address

City State Zip

Project Contact E-mail

Phone # Fax #

Sampler(s) Name(s) Phone #

Project Information

Project Name:

Street

City State

Project #

Client Purchase Order #

Project Manager

Billing Information (if different from Report to)

Company Name

Street Address

City State Zip

Attention

Requested Analysis (see TEST CODE sheet)

Matrix Codes

DW - Drinking Water
GW - Ground Water
WW - Water
SW - Surface Water
SO - Soil
SL- Sludge
SED-Sediment
OI - Oil
LIQ - Other Liquid
AIR - Air
SOL - Other Solid
WP - Wipe
FB-Field Blank
EB-Equipment Blank
RB- Rinse Blank
TB-Trip Blank

Accutest Sample #	Field ID / Point of Collection	MEOH/DI Vial #	Collection Date	Collection Time	Sampled by	Matrix	# of bottles	Number of preserved Bottles: HCl	NaOH	HNO3	H2SO4	NONE	DI Water	MEOH	ENCORE	LAB USE ONLY

Turnaround Time (Business days)

- ☐ Std. 15 Business Days
- ☐ Std. 10 Business Days (by Contract only)
- ☐ 10 Day *RUSH*
- ☐ 5 Day *RUSH*
- ☐ 3 Day *EMERGENCY*
- ☐ 2 Day *EMERGENCY*
- ☐ 1 Day *EMERGENCY*

Emergency & Rush T/A data available VIA Lablink

Approved By (Accutest PM): / Date:

Data Deliverable Information

- ☐ Commercial "A" (Level 1)
- ☐ Commercial "B" (Level 2)
- ☐ FULLT1 (Level 3+4)
- ☐ NJ Reduced
- ☐ Commercial "C"
- ☐ NYASP Category A
- ☐ NYASP Category B
- ☐ State Forms
- ☐ EDD Format ____
- ☐ Other ____

Commercial "A" = Results Only
Commercial "B" = Results + QC Summary
NJ Reduced = Results + QC Summary + Partial Raw data

Comments / Special Instructions

Sample Custody must be documented below each time samples change possession, including courier delivery.

Relinquished by Sampler:	Date Time:	Received By:	Relinquished By:	Date Time:	Received By:
1		1	2		2
Relinquished by Sampler:	Date Time:	Received By:	Relinquished By:	Date Time:	Received By:
3		3	4		4
Relinquished by:	Date Time:	Received By:	Custody Seal # ☐ Intact ☐ Not Intact	Preserved where applicable ☐	On Ice ☐ Cooler Temp.
5		5			

APPENDIX C

Accutest MDLS, RLS, and Control Limits

Control Limits (%) Rev: 6/12A

Compound	CAS No.	RL	MDL	Units	MS/MSD	RPD	BS	DUP
1,1,1-Trichloroethane	71-55-6	1	0.24	ug/l	61-146	14	81-131	10
1,1,2-Trichloroethane	79-00-5	1	0.29	ug/l	75-125	10	81-119	10
1,1-Dichloroethene	75-35-4	1	0.19	ug/l	52-142	15	75-124	10
1,2-Dichloroethane	107-06-2	1	0.26	ug/l	68-139	10	75-133	10
cis-1,2-Dichloroethene	156-59-2	1	0.19	ug/l	59-137	11	74-132	10
trans-1,2-Dichloroethene	156-60-5	1	0.21	ug/l	60-134	12	68-125	10
Ethylbenzene	100-41-4	1	0.23	ug/l	48-139	11	81-118	11
Tetrachloroethene	127-18-4	1	0.28	ug/l	57-144	13	60-159	10
Trichloroethene	79-01-6	1	0.22	ug/l	59-140	12	84-122	10
Vinyl chloride	75-01-4	1	0.21	ug/l	45-150	16	64-133	10
1,2-Dichloroethane-D4	17060-07-0				Surrogate Limits:		74-127	
4-Bromofluorobenzene	460-00-4				Surrogate Limits:		78-116	
Toluene-D8	2037-26-5				Surrogate Limits:		80-122	

APPENDIX D

Laboratory Standard Operating Procedures

Accutest

Lab Manager _____

QA Manager _____

Effective Date 4/8/13

TEST NAME: METHOD 8260B, VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/ MASS SPECTROMETRY (GC/MS)

METHOD REFERENCE: SW846 8260B (Revision 2, December 1996)

Revised Sections: Table 1 and Table 6.

1.0 SCOPE AND APPLICATION

1.1 This SOP describes the analytical procedures, which are utilized by Accutest to acquire samples for analysis of volatile organic compounds by gas chromatographic/mass spectrometric (GC/MS) following purge and trap utilizing the internal standard technique. The compounds in Table 1 may be determined by this method. An option has been included for the analysis of 1,4-Dioxane by selected ion monitoring GC/MS (GC/SIM-SIM).

1.2 This analytical method is designed for nearly all types of samples, regardless of water content, including ground water, aqueous sludges, liquors, waste solvents, oily wastes, tars, filter cakes, sediments and soils.

1.3 The applicable concentration range of this method is compound, matrix, and instrument dependent. Volatile water-soluble compounds can be included in this analytical technique. However, for some low-molecular weight halogenated hydrocarbons, aromatics, ketones, nitriles, acetates, acrylates, ethers, and sulfides, quantitation limits are approximately ten times higher because of poor purging efficiency. Determination of some structural isomers (i.e. xylenes) may also be hampered by coelution.

2.0 SUMMARY OF METHOD

2.1 Volatile compounds are introduced into the gas chromatograph by purge-and-trap (Method 5030/5035). Method 5030 may be used directly on ground water samples. Method 5035 is used for low-concentration and medium-concentration soils, sediments, and wastes. Medium concentration samples are preserved and stored in methanol prior to purge-and-trap analysis.

2.2 An inert gas is bubbled through a 5 ml sample contained in a specifically designed purging chamber at ambient temperature. The purgeables are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the purgeables are trapped. After purging is completed, the sorbent column is heated and backflushed with the inert gas to desorb the purgeables onto a gas chromatographic (GC) column.

2.3 The volatile compounds are separated by the temperature programmed GC column and detected using a mass spectrometer, which is used to provide both qualitative and quantitative information.

2.4 The peaks detected are qualitated by comparison to characteristic ions and retention times specific to the known target list of compounds.

2.5 Once identified the compound is quantitated by comparing the response of major (quantitation) ion relative to an internal standard technique with an average response factor generated from a calibration curve.

2.6 Additional unknown peaks with a response > 10 % of the closest internal standard may be processed through a library search with comparison to a database of approximately 75,000 spectra. An estimated concentration is quantitated by assuming a response factor of 1.

2.7 Water soluble volatile organic and other poor purging compounds maybe analyzed using this methodology, however this method is not the method of choice for these compounds and the laboratory's ability to achieve all calibration and quality control criteria for this method cannot be guaranteed. These compounds are noted as (pp) in Table 7.

2.8 The method includes an analytical option for the analysis of 1,4-Dioxane by GC/MS-SIM. The selected ions that are characteristic of the analytes of interest are analyzed using lower concentrations of calibration standards under the same MS conditions. SIM analysis is performed upon client request and is documented in the report.

3.0 REPORTING LIMIT AND METHOD DETECTION LIMIT

3.1 Reporting Limit. The reporting limit for this method is established at the lowest concentration standard in the calibration curve and may vary depending on matrix interferences, sample volume or weight and percent moisture. Detected concentrations below this concentration cannot be reported without qualification. See Table 10.

3.1.1 Compounds detected at concentrations between the reporting limit and MDL are quantitated and qualified as "J", estimated value. Program or project specifications may dictate that "J" qualified compounds are not to be reported.

3.2 Method Detection Limit. Experimentally determine MDLs using the procedure specified in 40 CFR, Part 136, Appendix B. This value represents the lowest reportable concentration of an individual compound that meets the method qualitative identification criteria.

3.2.1 Experimental MDLs must be determined annually for this method.

3.2.2 Process all raw data for the replicate analysis in each MDL study. Forward the processed data to the QA group for archiving.

4.0 DEFINITIONS

BLANK - an analytical sample designed to assess specific sources of laboratory contamination. See individual types of Blanks: Method Blank, Instrument Blank, Storage Blank, Cleanup Blank and Sulfur Blank.

4-BROMOFLUOROBENZENE (BFB) - the compound chosen to establish mass spectral instrument performance for volatile (VOA) analyses.

CALIBRATION FACTOR (CF) - a measure of the gas chromatographic response of a target analyte to the mass injected. The calibration factor is analogous to the Relative Response Factor (RRF) used in the Volatile and Semivolatile fractions.

CONTINUING CALIBRATION - analytical standard run every 12 hours to verify the initial calibration of the system.

CONTINUOUS LIQUID-LIQUID EXTRACTION - used herein synonymously with the terms continuous extraction, continuous liquid extraction, and liquid extraction. This extraction technique involves boiling the extraction solvent in a flask and condensing the solvent above the aqueous sample. The condensed solvent drips through the sample, extracting the compounds of interest from the aqueous phase.

EXTRACTED ION CURRENT PROFILE (EICP) - a plot of ion abundance versus time (or scan number) for ion(s) of specified mass (Es).

INITIAL CALIBRATION - analysis of analytical standards for a series of different specified concentrations; used to define the linearity and dynamic range of the response of the mass spectrometer to the target compounds.

INTERNAL STANDARDS - compounds added to every standard, blank, matrix spike, matrix spike duplicate, sample (for volatiles), and sample extract (for semivolatiles) at a known concentration, prior to analysis. Internal standards are used as the basis for quantitation of the target compounds.

MATRIX - the predominant material of which the sample to be analyzed is composed. For the purpose of this SOP, a sample matrix is either water or soil/sediment. Matrix is <u>not</u> synonymous with phase (liquid or solid).

MATRIX SPIKE - aliquot of a matrix (water or soil) fortified (spiked) with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

MATRIX SPIKE DUPLICATE - a second aliquot of the same matrix as the matrix spike (above) that is spiked in order to determine the precision of the method.

METHOD BLANK - an analytical control consisting of all reagents, internal standards and surrogate standards that is carried throughout the entire analytical procedure. The method blank is used to define the level of laboratory, background and reagent contamination.

METHOD DETECTION LIMITS (MDLs) - The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. MDLs should be determined approximately once per year for frequently analyzed parameters.

PERCENT DIFFERENCE (%D) - As used in this SOP and elsewhere to compare two values, the percent difference indicates both the direction and the magnitude of the comparison, i.e., the percent difference may be either negative, positive, or zero. (In contrast, see relative percent difference.)

PERCENT MOISTURE - an approximation of the amount of water in a soil/sediment sample made by drying an aliquot of the sample at 105°C. The percent moisture determined in this manner also includes contributions from all compounds that may volatilize at or below 105 °C, including water. Percent moisture may be determined from decanted samples and from samples that are not decanted.

PRIMARY QUANTITATION ION - a contract specified ion used to quantitate a target analyte.

REAGENT WATER - water in which an interferant is not observed at or above the minimum detection limit of the parameters of interest.

RECONSTRUCTED ION CHROMATOGRAM (RIC) - a mass spectral graphical representation of the separation achieved by a gas chromatograph: a plot of total ion current versus retention time.

RELATIVE PERCENT DIFFERENCE (RPD) - As used in this SOP and elsewhere to compare two values, the relative percent difference is based on the mean of the two values, and is reported as an absolute value, i.e., always expressed as a positive number or zero. (In contrast, see percent difference.)

RELATIVE RESPONSE FACTOR (RRF) - a measure of the relative mass spectral response of an analyte compared to its internal standard. Relative Response Factors are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples.

RELATIVE RETENTION TIME (RRT) - the ratio of the retention time of a compound to that of a standard (such as an internal standard).

INSTRUMENT BLANK – a system evaluation sample containing lab reagent grade water with internal standards and surrogate standards added. An instrument blank is used to remove and/or evaluate residual carryover from high level standards, spike samples and field samples.

5.0 HEALTH & SAFETY

5.1 The analyst must follow normal safety procedures as outlined in the Accutest Health and Safety Plan and Personal Protection Policy, which include the use of safety glasses and lab coats. In addition, all acids are corrosive and must be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor.

5.2 The toxicity or carcinogenicity of each reagent used in this method has not been precisely determined; however, each chemical must be treated as a potential health hazard. Exposure to these reagents must be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets must be made available to all personnel involved in these analyses.

5.3 The following analytes covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: benzene, carbon tetrachloride, 1,4-dichlorobenzene, 1,2-dichlorethane, hexachlorobutadiene, 1,1,2,2-tetrachloroethane, 1,1,2-trichloroethane, chloroform, 1,2-dibromoethane, tetrachloroethene, trichloroethene, and vinyl chloride. Primary standards of these toxic compounds must be prepared in a hood. A NIOSH/Mass approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

6.0 INTERFERENCES

6.1 The data from all blanks, samples, and spikes must be evaluated for interferences.

6.2 Impurities in the purge gas, organic compounds out-gassing from the plumbing ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks. The use of non-TFE tubing, non-TFE thread sealants, or flow controllers with rubber components in the purging device should be avoided.

6.3 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal into the sample during shipment and storage. A trip

blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

6.4 Contamination by carry-over can occur whenever high level and low-level samples are sequentially analyzed.

6.4.1 Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of an instrument blank to check for cross contamination. Refer to Table 11 for compounds that may cause carryover for this method.

6.4.2 It may be necessary to wash the purging device with methanol, rinse it with organic-free water, and then dry the purging device in an oven at 105^0 C. Follow the instrument manual for instructions on cleaning. Document the occurrence in the maintenance log and notify the manager/supervisor.

6.4.2.1 Clean and bake purging tube.

6.4.2.2 Clean or replace purge needle.

6.4.2.3 Clean and bake sample filter or sparge filter.

6.4.2.4 Clean and bake sample loop.

6.4.2.5 Replace trap if necessary.

6.4.2.6 Replace water management module if necessary.

6.4.2.7 Rinse transfer line with methanol. Caution: disconnect the trap before rinsing.

6.4.3 In extreme situations, the entire purge-and trap device may require dismantling and cleaning. Follow the instrument's manual for instructions on disassembly. Document the occurrence in the maintenance log and notify the manager/supervisor. Screening of the samples prior to purge-and-trap GC/MS analysis is highly recommended to prevent contamination of the system. This is especially true for soil and waste samples.

6.4.4 If the contamination has been transferred to gas chromatograph, any of the following approaches may be used to cleanup the instrument.

6.4.4.1 Baking out the column between analyses.

6.4.4.2 Change the injector liner to reduce the potential for cross-contamination.

6.4.4.3 Remove a portion of the analytical column in the case of extreme contamination.

6.4.5 The oven temperature program must include a post-analysis bake out period to ensure that semivolatile hydrocarbons are stripped from the chromatographic column.

6.4 Special precautions must be taken during the analysis to avoid contamination from methylene chloride and other common laboratory solvents.

6.5.1 The sample storage and analytical area should be isolated from all atmospheric sources of methylene chloride or other common solvents.

6.5.2 Laboratory clothing worn by the analyst should be clean and used in designated areas only. Clothing previously exposed to solvent vapors in the organics sample preparation laboratory can contribute to sample contamination.

7.0 SAMPLE HANDLING AND PRESERVATION AND HOLDING TIME

7.1 HANDLING and PRESERVATION

7.1.1 Water samples

7.1.1.1 Container - 40 ml glass screw-cap VOA vial with Teflon-faced silicone septum. The 40-ml glass VOA vials are pre-cleaned and certified.

7.1.1.2 Collect all samples in duplicate. Test all samples for residual chlorine using test paper for free and total chlorine. If samples contain residual chlorine, three milligrams of sodium thiosulfate should be added for each 40 ml of water sample.

7.1.1.3 Fill sample bottles to overflowing, but do not flush out the dechlorinating agent. Sample should be taken with care so as to prevent any air or bubbles entering vials creating headspace.

7.1.1.4 Adjust the pH of all samples to ≤ 2 at the time of collection, but after dechlorination, by carefully adding two drops of 1:1 HCl for each 40 ml of sample. Seal the sample bottles, Teflon face down, and mix for one minute. Or VOA vials containing the preservative(HCL) may be used.

Note: Do not mix the sodium thiosulfate with the HCl in the sample bottle prior to sampling.

7.1.1.5 The samples must be protected from light and refrigerated at 0 - ≤ 6 °C from the time of receipt until analysis.

7.1.2 Soil Samples

7.1.2.1 Refer to the SOP for SW846 Method 5035 for preservation requirement of non-aqueous solids. For Ohio VAP freezing is not allowed; samples must be preserved with sodium bisulfate.

7.2 HOLDING TIME

7.2.1 Water Samples.

7.2.1.1 All samples are to be analyzed within 14 days of sampling (HCl preserved for aqueous sample) unless otherwise specified by the contract. The sample preservation deficiency is noted in the analytical run logbook when the analyst checks the pH at the bench. If the pH is not <2, the analyst notifies the supervisor, who then notifies Client Service Dept. A comment is added to the result page and Non-Conformance Summary.

7.2.2 Soil Samples

7.2.2.1 Refer to the SOP for SW846 Method 5035 for holding time requirement of non-aqueous solids.

7.2.2.2 All samples are analyzed within 14 days of sampling unless otherwise specified.

8.0 APPARATUS AND MATERIALS

8.1 SYRINGE

8.1.1 10, 25, 50, 100, 500 and 5000 µl graduated syringes, manually held (Hamilton/equiv.).

8.1.2 5 ml and 50 ml glass gas tight syringes with Luerlok end, if appropriate for the purging device.

8.2 BALANCE

8.2.1 Analytical balance capable of weighing 0.0001 gram.

8.2.2 Top loading balance capable of weighing 0.1 gram.

8.3 PURGE AND TRAP DEVICES

8.3.1 The autosampler models are used for purging, trapping and desorbing the sample into GC column.

- O.I. Model 4560 sample concentrator with 4551 vial multi-sampler
- O.I. Model 4560 sample concentrator with 4552 Water/Soil multi-sampler

8.3.2 The sample purge vial must be designed to accept 5 ml samples with a water column at least 3 cm deep.

8.3.3 The auto-sampler is equipped with a heater capable of maintaining the purge chamber at 40 °C to improve purging efficiency. The heater is to be used for low level soil/sediment analysis, but not for water or medium level soil/sediment analysis.

8.3.4 The OI #10 trap is 42 cm with an inside diameter of 0.105 inches. The trap must be packed to contain the following absorbents (3-ring) and should be conditioned at 180 °C for 30 minutes by backflushing with a Helium gas flow at least 20 ml/min before initial use.

- Tenax (2,6-Diphenylene oxide polymer).
- Silica gel.
- Carbon Molecule Sieve (CMS).

8.3.5 The desorber should be capable of rapidly heating the trap to 190^0 C for desorption. Do not exceed $210\ ^0$C during bake-out mode. Alternatively, follow manufacturer's instructions.

8.3.6 The response of chloromethane and bromonethane can be tracked for thermal decomposition products formed. If levels over the calibration requirement, the trap must be replaced and the system re-calibrated after the manager/supervisor been notified.

8.4 GAS CHROMATOGRAPH/MASS SPECTROMETER SYSTEM

8.4.1 Gas Chromatograph.

8.4.1.1 An analytical system complete with a temperature programmable gas chromatograph and all required accessories including syringes, analytical columns, and gases.

8.4.1.2 The injection port should be suitable for split or splitless with appropriate interface.

8.4.1.3 The narrow bore capillary column is directly coupled to the source for HP-6890 model.

8.4.1.4 The wide bore capillary column is interfaced through a jet separator to the source for HP-5890 model.

8.4.2 Column.

- 75 m x 0.53mm ID x 3 µm film thickness capillary column coated with DB-624 (J&W Scientific), or equivalent. Condition as per manufactures directions.
- 105 m x 0.53mm ID x 3 µm film thickness capillary column coated with HP-VOA, or equivalent. Condition as per manufactures directions.
- 60 m x 0.25mm ID x 1.4 µm film thickness capillary column coated with DB-624 (J&W Scientific), or equivalent. Condition as per manufactures directions.
- 60 m x 0.45mm ID x 1.7 µm film thickness capillary column coated with DB-VRX (J&W Scientific), or equivalent. Condition as per manufactures directions.

8.4.3 Mass Spectrometer.

8.4.3.1 HP5973 or HP5970 is capable of scanning from 35 to 300 amu every 2 seconds or less, utilizing a 70 volt (nominal) electron energy in the electron impact ionization mode.

8.4.3.2 The mass spectrometer must be capable of producing a mass spectrum which meets all the criteria in Table 3 when injecting or purging 50 ng of the GC/MS tuning standard - Bromofluorobenzene (BFB).

8.4.3.3 SIM Mode – Capable of selective ion grouping at specified retention times for increased compound sensitivity (Table 2a).

8.5 DATA SYSTEM

8.5.1 Data Acquisition and Instrument Control (HP Chemstation) - A computer system is interfaced to the mass spectrometer, which allows the continuous acquisition and storage on a machine-readable media (disc) of all mass spectra obtained throughout the duration of the chromatographic program.

8.5.2 Data Processing (HP Enviroquant) - The software accommodates searching of GC/MS data file for target analytes which display specific fragmentation patterns. The software also allows integrating the abundance of an EICP between specified time or scan number limits. The data system includes the recent version of the EPA/NBS or NIST98 mass spectral library for qualitative searches of non-target compounds present in the chromatogram. The data system flags all data files that have been edited manually by laboratory personnel.

8.5.3 Off line Magnetic Tape Storage Device (Lagato Networker) - The magnetic tape storage device copies data for long-term, off-line storage.

9.0 REAGENTS AND STANDARDS

9.1 Solvent

9.1.1 Methanol: purge-and-trap grade quality or equivalent. Store separately, away from the other solvents.

9.2 Reagent Water

9.2.1 Reagent water is defined as water in which an interferant is not observed at the method detection limit of the parameters of interest.

9.2.2 Reagent water is generated by either passing tap water through a bed of approximately one pound of activated carbon or by using the water purification system at Accutest that is a series of deionizers and carbon cartridges.

9.3 Stock Standard Solutions

9.3.1 Commercially prepared standards used.

9.3.1.1 EPA Method 524.2 Volatiles (78 components): Absolute (or equivalent) at 200 µg/ml or 2,000 µg/ml concentration.

9.3.1.2 Custom Volatiles Mix A: Restek (or equivalent) at 2,000 µg/ml concentration.

9.3.1.3 Custom Volatiles Mix B: Restek (or equivalent) at 2,000 - 100,000 µg/ml concentration.

9.3.1.4 VOC Gas Mixture: Ultra (or equivalent) contains 200 µg/ml or 2,000 µg/ml of the following compounds in methanol.

- Bromomethane
- Chloroethane
- Chloromethane
- Dichlorodifluoromethane
- Trichlorofluoromethane
- Vinyl Chloride

9.3.1.5 Multiple neat compounds.

9.3.1.6 Surrogate standard mixture: Ultra (or equivalent) at a concentration of 2,500 µg/ml each surrogate compound.

- 1,2-Dichloroethane-d_4
- Dibromofluoromethane
- Toluene-d_8
- 4-Bromofluorobenzene

9.3.1.7 Internal standard mixture: Ultra (or equivalent) at a concentration of 2,000 µg/ml for all the compounds except Tert Butyl Alcohol-d_9, which is from Absolute (or equivalent) at a concentration of 50,000 µg/ml. The following five internal standards are used that exhibit similar analytical behavior to the compounds of interest.

- 1,4-Dichlorobenzene-d_4
- 1,4-Difluorobenzene
- Chlorobenzene-d_5
- Pentafluorobenzene
- Tert Butyl Alcohol-d_9

9.3.1.8 1,4-Dioxane Solution for SIMS : Ultra (or equivalent) at 100 µg/ml in methanol .

9.3.2 Unopened stock standard (ampoules) must be stored according to manufacturer's documented holding time and storage temperature recommendations (usually placed on the ampoule).

9.3.3 After opened, stock standards, internal standards, and surrogate solutions must be replaced after 6 months (one month for purgeable gases standard) or sooner if manufacture expiration date come first or comparison with quality control check samples indicates degradation.

9.3.4 Store all stock standards in vials with minimal headspace and Teflon lid liners after open, protect from light, and refrigerate to –10°C or colder or as recommended by the standard manufacturer.

9.3.5 Return the standards to the freezer as soon as the analyst has completed mixing or diluting the standards to prevent the evaporation of volatile target compounds.

9.4 Internal Standard and Surrogate Solution

9.4.1 Five internal standard and surrogate spiking solutions are prepared in methanol per Table 8.A.

9.4.1.1 25 µg /ml internal standard and surrogate mixture.

9.4.1.2 250 µg /ml internal standard and surrogate mixture.

9.4.1.3 100 µg/ml surrogate mixture.

9.4.1.4 25 µg /ml internal standard mixture.

9.4.1.5 250 µg /ml internal standard mixture.

9.4.2 A calibration range must be constructed for the surrogate compounds. Accordingly, appropriate amounts of surrogates are mixed with each calibration solution to define a range similar to the target compounds.

9.4.3 Each 5 ml sample, QC sample, and blank undergoing analysis should be spiked with any one of the above spiking solutions (depending upon the type of standards addition modules used), resulting in a concentration of 50 µg/l of each compound.

9.4.4 Prepare fresh internal standard and surrogate spiking solutions every six months, or sooner, if manufacturer's expiration dates come first or if the solution has degraded or evaporated.

9.5 Secondary Dilution Standards

9.5.1 Using stock standard solutions, prepare secondary dilution standards in methanol containing the compounds of interest, either singly or mixed together.

9.5.1.1 100 µg /ml V8260 mixture: prepared from 2,000 µg /ml stock solution. (see Table 8-C)

9.5.1.2 100 µg /ml V8260 custom mixture: prepared from 2,000 µg /ml stock solution. (see Table 8-C)

9.5.1.3 100 µg /ml Gas mixture: prepared from 2,000 µg /ml stock solution. (see Table 8-C)

9.5.2 Replace after one month for non-gas mixtures (one week for gas mixtures) or sooner if manufacture expiration date come first or comparison with quality control check samples indicates degradation.

9.5.3 Store all secondary dilution standards in vials with no headspace and Teflon lid liners, protect from light, and refrigerate to – 10°C or colder or according to manufacturer's storage temperature recommendation.

9.5.4 Return the standards to the freezer as soon as preparation is finished to prevent the evaporation of volatile compounds.

9.6 Aqueous Calibration Standard Solutions

9.6.1 Initial Calibration Standards

9.6.1.1 Prepare a minimum of five aqueous calibration standard solutions containing the surrogate compounds as Table 8-D.1 or 8-D.2.

9.6.1.2 To prepare a calibration standard, add a measured volume of secondary dilution standard solutions and the surrogate spiking solution to an aliquot of reagent water in the flask. Use a micro-syringe and rapidly inject the methanol standard into the expanded area of the filled volumetric flask. Remove the needle as quickly as possible after injection. Bring to volume. Mix by inverting the flask three times only. Discard the contents contained in the neck of the flask.

9.6.1.2.1 1,4-Dioxane for SIMS analysis is prepared from primary stock standard (100ppm).

9.6.2 Continuing Calibration Standard

9.6.2.1 A continuing calibration standard at a concentration of 50 µg/l is prepared as the scheme outlined in Table 8-E.

9.6.3 Aqueous standards are not stable and may be stored up to 24 hours if held in Teflon sealed screw-cap vials with zero headspace at 4^0C ($\pm$ 2^0C). Protect the standards from light. If not so stored, they must be discarded after use, unless they are set up to be purged by an autosampler.

9.6.4 When using an autosampler, standards may be retained up to 12 hours if they are in purge tubes connected via the autosampler to the purge and trap device.

9.7 Second Source Calibration Check Standard (ICV)

9.7.1 Prepare the second source calibration check standards from separate sources of stock standards from the calibration curve following the procedures in Section 9.6. At a minimum, an ICV should be analyzed with every initial calibration.

9.7.2 For 1,4-Dioxane via SIMS: Prepare the second source calibration check standard using 2.5 µl of a 1000ppm (Absolute or equivalent) to 50 mL of reagent water which yields a 50 ppb standard.

9.8 4-Bromofluorobenzene (BFB) Standard

9.8.1 Two BFB solutions are prepared in methanol per Table 8-B.

9.8.1.1 25 µg /ml solution for direct injection.

9.8.1.2 250 µg /ml solution for purging.

9.8.2 The solution must be replaced after 6 months or sooner if mass spectrum indicates degradation or if manufacture expiration date comes first.

10.0 CALIBRATION

10.1 Daily Maintenance. Routine Daily maintenance must be performed before any tuning, calibration or sample analysis activities are initiated. These include checks of the following items:

Purge and Trap Device:

Clean & bake purge tube
Bake trap and transfer lines
Check or refill internal/surrogate spike solution on SIM/SAM vials
Clean/replace syringe (if necessary)
Change and refill rinse bottle
Empty and rinse waste bottle

GC Oven: (if necessary)

Change septum
Change liner
Clip column, indicated by carbon build-up

10.2 Initial Calibration

10.2.1 The calibration range covered for routine analysis under RCRA, and SIM, employs standards of 1(specified compounds only), (2)*, 5, 10, 20, 50, 100, 200,(300 or 400)* µg/l. (*instrument dependent). A minimum of five standards must be run sequentially. The low calibration standard defines the reporting limit. Lower concentration standards (1.0 or 2.0 µg/l) may be needed to meet the reporting limit requirements of state specific regulatory programs. Refer to Table 8-D-1 and 8-D-2 for calibration standard preparation.

10.2.2 A calibration range must be constructed for each surrogate compound. Accordingly, add appropriate amounts of each surrogate compound to the calibration solution to define a range similar to the target compounds.

10.2.2.1 For most samples and spikes both the internal standard and the surrogate are added automatically. When doing an initial calibration surrogates are added manually. In order to compensate for the difference between the automatic and manual surrogate additions a correction factor must be applied to the amount of surrogate added in Table 8-D. To determine the correction factor divide the surrogate concentration from an automatic injection by the surrogate concentration from a manual injection for each of the surrogates. Average the result for each of the surrogates to determine the correction factor. Finally multiply the correction factor by the appropriate amount of surrogate from Table 8-D and add this amount to the standard.

10.2.3 For water and medium-level soil calibration: Transfer and fill up (no air space) each standard to labeled 40 ml vial and cap with Teflon septum, then place the vial into O.I. sample tray.

10.2.4 For low-level soil calibration: Transfer 5 ml of each standard to labeled 40 ml vial and cap with Teflon septum, then place the vial into O.I. sample tray.

10.2.4.1 When calibrating for Method 5035 low-level samples, if the sodium bisulfate option was used, add 1g of sodium bisulfate to the 40-ml vial before aliquot 5 ml of each standard into vial otherwise do not add sodium bisulfate. This is equivalent to the amount of sodium bisulfate added to the samples and will maintain a consistent purging efficiency of the compounds. Cap the vial with Teflon septum and place it into O.I sample tray.

10.2.5 The linear range covered by this calibration is the highest concentration standard.

10.2.6 Program the autosampler to add internal standard mixture to each standard. This results in a concentration of 50 µg/l for each internal standard.

10.2.6.1 For O.I. SIM spiker: Automatically adds 10 µl of 25 µg/ml internal standard solution (Section 9.4.1.4) to each standard.

10.2.6.2 For O.I. SAM spiker: Automatically adds 1 µl of 250 µg/ml internal standard solution (Section 9.4.1.5) to each standard.

10.2.7 Analyze the standard solutions using the conditions established in Section 11.0. Whenever the highest concentration standard is analyzed, it is usually followed by the analyses of two reagent water blanks. Further analysis may not proceed until the blank analysis is demonstrated to be free of interferences.

10.2.8 Each analyte is quantitatively determined by internal standard technique using the closest eluting internal standard and the corresponding area of the major ion. See Table 7.

10.2.9 The Response Factor (RF) is defined in Section 13.1. Calculate the mean RF for each target analyte using minimum of five RF values calculated from the initial calibration curve.

10.2.10 For the initial calibration to be valid, the following criteria must be met.

10.2.10.1 Five compounds (System Performance Check Compounds, SPCCs) are checked for a minimum average response factor. The minimum mean response factors are listed in Table 6. If the initial calibration criteria for SPCCs are not achieved, perform corrective action before completing the calibration

10.2.10.2 The % RSD for each individual Calibration Check Compound (CCC) must be less than 30 %. This check is used to identify gross instrument operating problems. If the initial calibration criteria for CCCs are not achieved, perform corrective action before completing the calibration.

10.2.10.3 The percent relative standard deviation (% RSD) (see Section 13.2) of all target analytes must be less than 15 %.

10.2.10.4 If the average response factor criteria cannot be achieved, and if the problem is associated with one or more of the standards, reanalyze the standards and recalculate the RSD. The instrument logbook should have clear documentation as to what the suspected problem was.

10.2.10.4.1 A calibration standard is allowed to be repeated only once; if the second trial fails, a new initial calibration must be performed. Notify the team leader/manager. Document this occurrence in the instrument log.

10.2.10.5 Alternately, if the average response factor criteria cannot be achieved, the calibration range can be narrowed by dropping the low or high point of the curve.

10.2.10.5.1 The changes to the upper end of the calibration range will affect the need to dilute samples above the range, while changes to the lower end will affect the overall sensitivity of the method. Consider the regulatory limits or action levels associated with the target analytes when adjusting the lower end.

10.2.10.6 If the average response factor criteria still cannot be achieved, employ an alternative calibration linearity model. Specifically, linear regression using a least squares approach may be employed.

10.2.10.6.1 If Linear regression is employed select the linear regression calibration option of the mass spectrometer data system. Do not force the regression line through the origin and do not employ 0,0 as a sixth calibration standard.

10.2.10.6.2 The correlation coefficient (r value) must be ≥0.99 for each compound to be acceptable.

10.2.10.6.3 Perform corrective action and recalibrate if the calibration criteria cannot be achieved.

10.2.10.7 The initial calibration criteria for this method applies to all additional compounds of concern specified by the client.

10.2.10.8 The relative retention times of each target analyte in each calibration standard should agree within 0.06 relative retention time units.

10.3 Initial Calibration Verification (ICV) - Second Source Calibration Check Standard

10.3.1 The calibration is verified with a calibration check standard at 50 µg/l from an external source (Section 9.7). It must be analyzed immediately following the initial calibration.

10.3.2 The percent difference (% D) (Section 13.3) for this standard must meet the criteria of 20% for all the target compounds.

10.3.2.1 If % D is greater than 20%, reanalyze the second source check. If the criteria cannot be met upon re-injection, re-prepare the second source solution using a fresh ampoule and repeat the process.

10.3.2.2 If the %D criteria cannot be achieved after re-preparation of the second source, prepare a third source and repeat the process. Make fresh calibration standards using one of the two standard sources that matches each other and repeat the initial calibration.

10.4 Continuing Calibration Verification Standard(CCV)

10.4.1 A continuing calibration verification standard at a concentration near mid-level of the initial calibration range (50 µg/l) must be acquired every 12 hrs or at the beginning of each analytical batch.

10.4.1.1 For water and medium level soil analysis: Transfer and fill up (no air space) the calibration verification standard to labeled 40 ml vial and cap with Teflon septum, then place the vial into O.I. sample tray. Analyze as per Section 11.7.

10.4.1.1.1 Vary the concentration of the continuing calibration verification standard on alternate verifications (i.e. every other calibration verification) using an alternative concentration standard. The standard selected must be lower than the midpoint calibration standard.

10.4.1.2 For low-level soil analysis: Transfer 5 ml of the calibration verification standard to labeled 40 ml vial and cap with Teflon septum, then place the vial into O.I. sample tray. Analyze as per Section 11.7.

10.4.1.2.1 When calibrating for Method 5035 low-level samples, if the sodium bisulfate option was used add 1g of sodium bisulfate to the 40-ml vial before aliquot 5 ml of the calibration verification standard into vial, otherwise do not use sodium bisulfate. This is equivalent to the amount of sodium bisulfate added to the samples and will maintain a consistent purging efficiency of the compounds. Analyze as per Section 11.7.

10.4.1.3 A continuing calibration standard is analyzed whenever the analyst suspects that the analytical system is out of calibration. If the calibration cannot be verified, corrective action is performed to bring the system into control. Analysis may not continue until the system is under control.

10.4.2 For the continuing calibration to be valid, all of the following specified criteria must be met.

10.4.2.1 The minimum RF for SPCC compound is shown on Table 6. Each SPCC compound in the calibration verification standard must meet its minimum response factor.

10.4.2.2 The percent difference (% D, see Section 13.3) for CCC must be less than 20%.

10.4.2.2.1 If the CCCs and SPCCs are not required analytes, such as the shortlist analysis for BTEX only, then all required project analytes must meet the 20 %D.

10.4.3 If the first continuing calibration verification(CCV) does not meet criteria, a second consecutive standard can be analyzed immediately. If the second CCV fails to meet criteria then corrective actions shall be performed. Such as: auto-tuning, routine system cleaning and routine system maintenance. Notify the team leader/manager.

10.4.3.1 If the second CCV trial fails, the lab must demonstrate acceptable performance after corrective action with two consecutive passing calibration verifications(CCVs) OR a new initial calibration. The Instrument Logbook and Maintenance Logbook must have clear documented notations as to what the problem was and what corrective action was implemented.

10.4.3.1.1 If the lab has not verified calibration, samples cannot be analyzed.

10.4.3.1.2 However, in the case where samples are analyzed on the system where the CCV does not meet the criteria the data must be flagged.

10.4.3.1.2.1 The data may be usable if the response for the verification exceed high(high bias) and the associated samples are non-detects.

10.4.3.1.2.2 If the criteria for the CCV is low (low bias), those sample results may be reported only if they exceed a maximum regulatory limit/decision level.

10.4.3.2 If the calibration verification is being performed using an auto sampler for night batch, two (2) vials of standard solution are placed in the device for analysis. The second standard must meet continuing calibration criteria and is used for calibration verification. The second check may be discarded only if there is a purge failure or incorrect spike concentration provided the first calibration standard meets the requirement. In this case, the first calibration standard is used as calibration verification following team leader/manager approval. Document this occurrence on instrument log.

10.4.3.2.1 Both CCVs must be evaluated. If vial 1 fails and vial 2 passes this meets the criteria of 10.4.3 of consecutive and immediate passing CCV.

10.4.3.2.2 If CCV number 2 fails, the analysis cannot continue unless it was determined that there was an isolated mechanical failure.

10.4.4 If any of the internal standard areas change by a factor of two (- 50% to + 100%) or the retention time changes by more than 30 seconds from the midpoint standard of the last initial calibration, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate.

10.4.5.1 Reanalyze the continuing calibration standard. New initial calibration is required if reanalyzed standard continues to fail the internal standard requirements.

10.4.5.2 All samples analyzed while the system was out of control must be reanalyzed following corrective action.

10.5 Corrective Action Maintenance For Failed Tuning and Calibration Procedures

10.5.1 Inability to achieve criteria for instrument tuning or calibration may indicate the need for instrument maintenance. Maintenance may include routine system cleaning and replacement of worn expendables or the need for outside service if the scope of the repair exceeds the capability of the staff.

10.5.2 If maintenance is performed on an instrument, return to control must be demonstrated before analysis can continue. Return to control is demonstrated as follows:

10.5.2.1 Successful instrument tune using PFTBA.

10.5.2.2 Successful tune verification by the analysis of 4-bromofluorobenzene.

10.5.2.3 Successful initial calibration or continuing calibration.

11.0 PROCEDURE

11.1 Instrument conditions.

11.1.1 Recommended instrument conditions are listed in Table 2 and 2a (SIM only). Modifications of parameters specified with an asterisk are allowed as long as criteria of calibration are met. Any modification should be approved by team leader/manger.

11.1.2 Optimize GC conditions for analyte separation and sensitivity. Once optimized, use the same GC conditions for the analysis of all standards, blanks, samples, and QC samples.

11.2 Purge and Trap Device conditions.

11.2.1 See Table 2.

11.2.2 Daily Maintenance. Routine Daily maintenance must be performed before any tuning, calibration or sample analysis activities are initiated. These include checks of the following items:

Purge and Trap Device:

- Clean & bake purge tube.
- Bake trap and transfer lines.
- Check or refill internal/surrogate spike solution on SIM/SAM vials.
- Clean/replace syringe (if necessary).
- Change and refill rinse bottle.
- Empty and rinse waste bottle.

11.3 Step 1: Daily GC/MS performance check.

11.3.1 Every 12 hours, either

- Inject 2 µl (50 ng) of BFB solution directly on column or
- Purge 10 µg/l of 5ml (50ng) to GC column.

11.3.2 The GC/MS system must be checked to verify acceptable performance criteria are achieved (see Table 3).

11.3.3 This performance test must be passed before any samples, blanks or standards are analyzed. Evaluate the tune spectrum using three mass scans from the chromatographic peak and a subtraction of instrument background.

11.3.3.1 Select the scans at the peak apex and one to each side of the apex.

11.3.3.2 Calculate an average of the mass abundances from the three scans.

11.3.3.3 Background subtraction is required. Select a single scan in the chromatogram that is absent of any interfering compound peaks and no more than 20 scans prior to the elution of BFB. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the tuning compound peak.

11.3.4 If all the criteria are not achieved, the analyst must retune the mass spectrometer with team leader/manager and repeat the test until all criteria are met.

11.3.4.5 Alternatively, an additional scan on each side of the peak apex may be selected and included in the averaging of the mass scans. This will provide a mass spectrum of five averaged scans centered on the peak apex. NOTE: The selection of additional mass scans for tuning may only be performed with supervisory approval on a case by case basis.

11.3.5 The injection time of the acceptable tune analysis is considered the start of the 12-hour clock.

11.3.6 Until performance check is acceptable, then calibration check (step 2) can be analyzed.

11.4 Step 2 : Daily calibration check

11.4.1 Initial calibration

11.4.1.1 Refer to Section 10.2.

11.4.1.2 An initial calibration must be established (or reestablished) on each instrument:

- Prior to any sample analyses;
- Whenever a new column is installed;
- Whenever instrument adjustments that affect sensitivity are made; and
- Whenever a continuing calibration standard fails to meet the specified acceptance criteria, on the second trial.

11.4.2 Initial Calibration Verification - Second Source Calibration Check Standard

11.4.2.1 This standard is only analyzed when initial calibration provided. Refer to Section 10.3.

11.4.3 Continuing Calibration verification standard

11.4.3.1 Refer to Section 10.4.

11.4.4 The method blank (step 3) cannot be analyzed until the continuing calibration verification meets the criteria.

11.5 Step 3 : Method blank

11.5.1 The acceptable method blank must be analyzed for every 12-hour time period or sooner.

11.5.1.1 Water and medium-level soil samples - Place a 40 ml vial, filled with DI water onto the autosampler.

11.5.1.2 Low-level soil samples without sodium bisulfate - Transfer 5 ml of DI water to a 40 ml vial and cap with Teflon septum, then place the vial into O.I. sample tray.

11.5.1.2.1 Low-level soil samples with sodium bisulfate (Method 5035) - Add 1g of sodium bisulfate to a 40 ml vial before aliquot 5 ml of DI water into the vial and cap with Teflon septum, then place the vial onto the autosampler.

11.5.2 Program the autosampler to add internal standard and surrogate solution to the method blank for a concentration of 50 µg/l for each internal standard and surrogate.

11.5.2.1 For O.I. SIM spiker: Automatically adds 10 µl of 25 µg/ml internal standard and surrogate solution (Section 9.4.1.1) to the method blank.

11.5.2.2 For O.I. SAM spiker: Automatically adds 1 µl of 250 µg/ml internal standard and surrogate solution (Section 9.4.1.2) to the method blank.

11.5.3 No compound can be present above the laboratory's MDL. If common laboratory solvents (i.e. methylene chloride, acetone) are present in the sample between the MDL and RL, the analyst must determine if the contamination will negatively impact data quality. If the contamination impacts data quality, all affected samples must be re-analyzed.

11.5.4 Surrogates must meet recovery criteria specified in house limits.

11.5.5 If the method blank does not meet surrogate criteria or contains target analytes above the MDL, then

11.5.5.1 All samples analyzed following an out of control method blank must be reanalyzed.

11.5.5.2 Check for the potential of contamination interference from the following areas. Make sure all items are free contamination.

- the analytical system,
- dust and vapor in the air,
- glassware and
- Reagents.

11.5.5.3 Re-analyze the method blank following the system evaluation. In this situation, the instrument logbook should have clear documented notations as to what the problem was and what corrective action was implemented to enable the second blank to pass.

11.5.5.4 If re-analyzed method blank remains out of control, notify team leader or manager.

11.5.6 If two consecutive method blanks are analyzed during unattended operations, the second analysis must meet criteria for the subsequent sample analysis to be valid. Always report the second method blank. The second analysis can only be discarded because of a purge failure provided that the first blank meets the requirement. In this case, the first blank is reported following team leader/manager approval. Document this occurrence on the instrument log.

11.5.7 The blank spike (BS) (step 4) cannot be analyzed until the method blank meets criteria.

11.6 Step 4: Blank spike (BS)

11.6.1 An acceptable blank spike must be analyzed with every analytical batch. The maximum number of samples per analytical batch is twenty.

11.6.2 Spike 50 ml of reagent water with appropriate amount of the standards to prepare a blank spike containing 50 µg/L of each analyte. In situations where lower detection limits are required, a blank spike at 20 µg/L may be prepared. The stock solution for the BS must be from a different source than the initial calibration solution. Refer to Table 8-F for the preparations of the blank spikes.

11.6.2.1 Water and medium-level soil samples - Place a 40 ml vial, filled with DI water onto the autosampler.

11.6.2.2 Low-level soil samples without sodium bisulfate - Aliquot 5 ml of the blank spike into vial and cap with Teflon septum, then place the vial into O.I. sample tray.

11.6.2.2.1 Low-level soil samples with sodium bisulfate for Method 5035 - Add 1g of sodium bisulfate to labeled 40 ml vial before aliquot 5 ml of the blank spike into vial and cap with Teflon septum, then place the vial into O.I. sample tray.

11.6.3 Initiate auto addition of internal standard and surrogate into the syringe per 11.5.2.

11.6.4 Compare the percent recoveries (% R) (see Section 13.5) to the in house limits acceptance criteria. If a blank spike is out of control, all the associated samples must be reanalyzed. The exception is if the blank spike recovery is high and no hits reported in associated samples and QC batch. In that case, the sample results can be reported with footnote (remark) and no further action is required. Or if the blank spike recovery is low and the hits in the samples are above regulatory levels.

11.6.5 Do not analyze samples and MS/MSD (step 5) unless the BS meets acceptance criteria.

11.7 Step 5: Samples /MS/MSD analysis

11.7.1 All samples and standard solutions must be allowed to warm to ambient temperature before analysis.

11.7.2 Select the sample dilution factor to assure the highest concentration analyte is above the calibration range midpoint, but below the upper limit of the range depend on project requirements. See Table 9 for dilution guideline.

- Utilize FID screen data.
- Utilize acquired sample data.
- Utilize the history program.
- Sample characteristics (appearance, odor).

11.7.3 Water samples.

11.7.3.1 Using O.I.Model 4560 sample concentrator with 4551 or 4552 vial multisampler,

- Place the 40 ml vial in the tray, or
- Load 5ml sample into purge tube if sample volume limited.

11.7.4 Sediment/ soil sample

11.7.4.1 Low-level soil method

11.7.4.1.1 Collect the sample using the procedures detailed in the SOP for SW846 Method 5035 low - level soil samples.

11.7.4.1.2 Weigh out 5 g of each sample into a labeled vial. Add 5 ml of reagent water and cap the vial quickly. Transfer the 40ml vial to the autosampler tray. Stir and heat the sample at the time of analysis.

11.7.4.2 Medium-level soil method

11.7.4.2.1 Collect the sample using the procedures detailed in the SOP for SW846 Method 5035 medium - level soil samples.

11.7.4.2.2 Select a methanol aliquot of appropriate volume (see Table 9) determined via screening and transfer to 40 ml of reagent water.

11.7.5 Program the autosampler to inject the internal standard and surrogate solution into the robotic syringe used to withdraw sample from the 40 ml vial. This addition to 5 ml of sample is equivalent to a concentration of 50 µg/L of each internal standard and surrogate.

11.7.5.1 For O.I. SIM spiker: Automatically adds 10 µl of 25 µg/ml internal standard and surrogate solution (Section 9.4.1.1) to each sample.

11.7.5.2 For O.I. SAM spiker: Automatically adds 1 µl of 250 µg/ml internal standard and surrogate solution (Section 9.4.1.2) to each sample.

11.7.6 Purge the sample for 11 minutes with Helium.

11.7.6.1 Low-level soil sample must be performed at 40 °C while the sample is being agitated with the magnetic stirring bar or other mechanical means.

11.7.6.2 To improve the purging efficiency of water-soluble compounds, aqueous samples may also be purged at 40 °C as long as all calibration standards, samples and QC samples are purged at the same temperature and acceptable method performance is demonstrated.

11.7.7 One sample is randomly selected from each analytical batch of similar matrix types and spiked in duplicate to determine whether the sample matrix contributes bias to the analytical results. A matrix spike and matrix spike duplicate are performed by spiking the sample for a concentration of 50 µg/l or 50 µg/kg based on 5 g dry weight. In situations where lower detection limits are required, a blank spike at lower concentration may be prepared.

11.7.8 Desorb the sample for 4 minutes by rapidly heating the trap to 190 °C while backflushing with Helium. Desorb time may require performance optimum between 2.0 and 4.0 minutes as dictated by trap manufacturers specifications or instrument characteristics.

11.7.9 Program the purge and trap system to automatically rinse purge tube at least twice with heated organic-free water (reagent water) between analyses to avoid carryover of target compounds. For samples containing large amounts of water-soluble materials, suspended solids, high-boiling compounds, or high purgeable levels, it may be necessary to wash out the purging device with methanol solution between analyses, rinse it with distilled water.

11.7.10 Bake the trap at least 10 minutes at 210 °C to remove any residual purgeable compounds.

11.7.11 If the initial analysis of the sample or a dilution of the sample has a response for any ion of interest that exceeds the working range of the GC/MS system, the sample must be reanalyzed at a higher dilution.

11.7.11.1 When ions from a compound in the sample saturate the detector, this analysis must be followed by the analysis of reagent water blank. If the blank analysis is not free of interferences, then the system must be decontaminated. Sample analysis may not resume until the blank analysis is demonstrated to be free of interferences.

11.8 Sample dilutions

11.8.1 Using Screening Data to Determine Dilution Factors

11.8.1.1 Dilution for High Concentration Analytes Exceeding The Calibration Range

11.8.1.1.1 The highest concentration target compound detected in the screen data is compared to the highest concentration calibration standard used for determinative volatile organics analysis.

11.8.1.1.1.1 Divide the calibration concentration of the highest concentration calibration standard by the screen concentration.

11.8.1.1.1.2 If the result is >1, sample dilution is considered.

11.8.1.1.2 The result from step 11.8.1.1.1 determines the dilution factor. The dilution factor is targeted to assure that the highest concentration diluted analyte is at the mid-range concentration of the calibration curve for the determinative analysis.

11.8.1.1.3 In all cases a conservative approach to dilution is applied to minimize the increase of detection and reporting limits

11.8.1.2 Dilution for High Concentration Matrix Interferences

11.8.1.2.1 The peak height of the background is compared to the peak height of the later eluting calibration standards from the screening analysis.

11.8.1.2.1.1 A rough estimate of background concentration is calculated by dividing the background peak height by the peak height of the selected screening standard and multiplying by its concentration.

11.8.1.2.2 If the result is >1, sample dilution is considered.

11.8.1.2.3 The result from step 11.8.1.2.1 determines the dilution factor. The dilution factor is targeted to avoid Carry-over contamination between samples and facilitate qualitative and quantitative analysis of target compounds present in the sample.

11.8.1.2.4 In all cases a conservative approach to dilution is applied to minimize the increase of detection and reporting limits

11.8.2 If the concentration of any target compound in any sample exceeds the initial calibration range, a new aliquot of that sample must be diluted and re-analyzed. Until the diluted sample is in a sealed sample vial, all steps in the dilution procedure must be performed without delay.

11.8.3 Water Samples.

11.8.3.1 Prepare all dilutions of water samples in volumetric flasks (10 ml to 100 ml). Intermediate dilutions may be necessary for extremely large dilutions.

11.8.3.2 Calculate the approximate volume of reagent water, which will be added to the volumetric flask, and add slightly less than this quantity to the flask. Refer to Table 9 for dilution guideline.

11.8.3.3 Inject the proper sample aliquot from a syringe into the volumetric flask. Dilute the flask to the volume mark with reagent water. Cap the flask and invert the flask three times.

11.8.3.4 Fill a 40 ml sample vial and seal with a Teflon baked silicon septa, load the diluted sample into the autosampler and analyze according to Section 11.7.

11.8.3 Low-level Soil Samples.

11.8.3.1 The screening data are used to determine which is the appropriate sample preparation procedure for the particular sample, the low-level soil method or the medium-level soil method.

11.8.3.2 If any target compound exceeds the initial calibration range from the analysis of 5 g sample; a smaller sample size must be analyzed. However, the smallest sample size permitted is 0.5 g. If smaller than 0.5 g sample size is needed to prevent any target compounds from exceeding the initial calibration range, the medium level method must be used.

11.9 Data interpretation

11.9.1 Qualitative identification.

11.9.1.1 The targeted compounds shall be identified by analyst with competent knowledge in the interpretation of mass spectra by comparison of the sample mass spectrum to the mass spectrum of a standard of the suspected compound.

11.9.1.2 The characteristic ions for target compounds that can be determined are listed in Table 7. Table 4 and Table 5 list the characteristic ions for internal standards and surrogate compounds respectively.

11.9.1.3 The criteria required for a positive identification are listed below.

11.9.1.3.1 The sample component must elute at the same relative retention time (RRT) as the daily standard. Criteria are the RRT of sample component must be within ± 0.06 RRT units of the standard component.

11.9.1.3.2 The relative intensities of these ions must agree within ± 30 % between the daily standard and sample spectra. (Example: For an ion with an abundance of 50 % in the standard spectra, the corresponding sample abundance must be between 20 and 80 %.)

11.9.1.3.2.1 Compounds can have secondary ions outside criteria from co-eluting compounds and/or matrix effect that can contribute to ion abundances. The interference on ion ratios can't always be subtracted out by software programs resulting in qualified compound identification.

11.9.1.3.2.2 Quantitation reports display compounds that have secondary ions outside the ratio criteria with a "#" flag.

11.9.1.3.2.3 Any quant reports with compounds that are deemed to be reportable despite the "#" flag, will be initialed in the "#" column by the analyst. Further review to the reporting of qualified compounds will be done by a supervisor or team leader and initialed on the quantitation.

11.9.1.3.3 Structural isomers that produce very similar mass specrtra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25 % of sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

11.9.2 Quantitative analysis

11.9.2.1 Once a target compound has been identified, its concentration (Section 13.4) will be based on the integrated area of the quantitation ion, normally the base peak (Table 7). The compound is quantitated by internal standard technique with an average response factor generated from the initial calibration curve.

11.9.2.2 If the sample produces interference for the primary ion, use a secondary ion to quantitate (see Table 7). This is characterized by an excessive background signal of the same ion, which distorts the peak shape beyond a definitive integration. Also interference could severely inhibit the response of the internal standard ion. This secondary ion must also be used to generate new calibration response factors.

11.10 Library search for tentatively identified compounds.

11.10.1 If a library search is requested, the analyst should perform a forward library search of NBS or NIST98 mass spectral library to tentatively identify 15 non-reported compounds.

11.10.2 Guidelines for making tentative identification are listed below.

11.10.2.1 These compounds should have a response greater than 10 % of the nearest internal standard. The response is obtained from the integration for peak area of the Total Ion Chromatogram (TIC).

11.10.2.2 The search is to include a spectral printout of the 3 best library matches for a particular substance. The results are to be interpreted by analyst.

11.10.2.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

11.10.2.4 Relative intensities of major ions in the reference spectrum (ions > 10 % of the most abundant ion) should be present in the sample spectrum.

11.10.2.5 The relative intensities of the major ions should agree within ± 20 %. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must between 30 and 70%).

11.10.2.6 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

11.10.2.7 Ions present in the reference spectrum but not in the sample spectrum should be verified by performing further manual background subtraction to eliminate the interference created by coeluting peaks and/or matrix interference.

11.10.2.8 Quantitation of the tentatively identified compounds is obtained from the total ion chromatogram based on a response factor of 1 and is to be tabulated on the library search summary data sheet.

11.10.2.9 The resulting concentration should be reported indicating: (1) that the value is estimate, and (2) which internal standard was used to determine concentration. Quantitation is performed on the nearest internal standard.

11.11 An instrument blank is a system evaluation sample containing lab reagent grade water with internal standards and surrogates. An instrument blank is used to remove and or evaluate residual carryover from high level standards, spike samples and field samples. Since target compound lists have expanded to overlap some volatile and semi-volatile compounds, instrument blanks are necessary to remove carryover contamination.

11.11.1 The compounds that may exhibit carryover for this method are listed in Table 11.

11.11.2 If instrument blanks following a standard or spike sample exhibits carrry-over effect, then any samples that show the same carryover profile, after a comparable concentration must be considered suspect and rerun for confirmation. For example, if an instrument blank has 1ppb detected after a 200ppb standard, then any sample following a sample containing 200ppb or above of the same compound must be confirmed for possible carryover.

11.11.3 If an Instrument Blank(s) was run following suspect high concentration samples and it exhibits the same carryover profile after a comparable concentration must be considered suspect and rerun for confirmation.

11.11.4 In some cases, several instrument blanks may have to be run to eliminate contamination from over loaded samples.

11.11.5 The analytical system is considered free of carryover, when no target analytes can be detected above the MDL.

11.12 Selected Ion Monitoring (SIM) Option Selected Ion Monitoring (SIM) Option

11.12.1 Instrument Set-Up: Modify the method for SIM analysis and define ion groups with retention times, ions and dwell times to include base peak ion for the target compounds of interest, surrogates, and internal standards (Table 2a.) Select a mass dwell time of 50 milliseconds for all compounds.

11.12.2 Calibration: Calibrate the mass spectrometer in the selected ion monitoring mode using 6 calibration standards of 5, 10, 20, 50, 100, 200 ug/l. Spike each standard with the SIM specific internal standard solution at 4ug/ml. Calculate individual response factors and response factor RSDs.

11.12.3 Initial Calibration Verification. Verify the initial calibration after its completion using a 50 ug/l calibration standard purchased or prepared from a second standards reference materials source. The initial calibration verification must meet the criteria of Section 10.2.

11.12.4 Continuing Calibration Verification. Verify the initial calibration every 12 hours using a 50 ug/l calibration. The continuing calibration verification must meet the criteria of Section 10.4.

11.12.5 Surrogate Standard Calculation.. Report surrogate spike accuracy for the surrogates spiked for the full scan GC/MS analysis.

12.0 QUALITY CONTROL

12.1 QC Requirements Summary

BFB	Beginning of the analytical shift and every 12 hours
ICV - Second Source Calibration Check Standard	Following initial calibration
Calibration Verification Standard	Every 12 hours
Method Blank	Every 12 hours
Blank Spike	One per analytical batch*
Matrix Spike	One per analytical batch*
Matrix Spike Duplicate	One per analytical batch*
Surrogate	Every sample and standard
Internal Standard	Every sample and standard

*The maximum number of samples per analytical batch is twenty.

12.2 Daily GC/MS Performance Check - BFB

12.2.1 Refer to Section 11.3.

12.3 Second Source Calibration Check Standard

12.3.1 Refer to Section 10.3.

12.3.2 Calibration Verification Standard

12.3.3 Refer to Section 10.4.

12.5 Method Blank

12.5.1 Refer to Section 11.5.

12.6 Blank Spike

12.6.1 Refer to Section 11.6.

12.7 Matrix Spike (MS)/Matrix Spike Duplicate (MSD)

12.7.1 One sample is selected at random from each analytical batch of similar matrix types and spiked in duplicate to check precision and accuracy.

12.7.2 Assess the matrix spike recoveries (Section 13.5) and relative percent difference (RPD) (Section 13.6) against the control limits..

12.7.3 If the matrix spike recoveries do not meet the criteria, check the blank spike recovery to verify that the method is in control. If the blank spike did not meet criteria, the method is out of control for the parameter in question and should be reanalyzed or qualified with an estimate of potential bias. Otherwise, matrix interference is assumed and the data is reportable. No further corrective action is required.

12.8 Surrogates

12.8.1 All standards, blanks, samples, and matrix spikes contain surrogate compounds, which are used to monitor method performance. If the recovery of any surrogate compound does not meet the control limits, the result must be flagged and:

12.8.1.1 The calculation must be checked.

12.8.1.2 The sample must be reanalyzed if the recovery of any one surrogate is out of control limit.

12.8.2 If the sample exhibits matrix interference, defined as excessive signal levels from target or non-target interfering peaks. In this case, reanalysis may not be required following team leader/manager approval.

12.8.3 If surrogate recoveries are acceptable upon reanalysis, the data from the reanalysis is reported. If the reanalysis date did not meet the hold time, then both sets of data must be submitted with the reanalysis reported.

12.8.4 If surrogates are still outside control limits upon reanalysis, then both sets of data should be submitted with the first analysis reported.

12.9 Internal Standard

12.9.1 Retention time for all internal standards must be within ± 30 seconds of the corresponding internal standard in the latest continuing calibration or 50 µg/l standard of initial calibration.

12.9.2 The area (Extracted Ion Current Profile) of the internal standard in all analyses must be within 50 to 200 % of the corresponding area in the latest calibration standard (12 hr. time period).

12.9.3 If area of internal standard does not meet control limits, the calculations must be checked. If a problem is not discovered, the sample must be reanalyzed.

12.9.4 If areas are acceptable upon reanalysis, the reanalysis data is reported.

12.9.5 If areas are unacceptable upon reanalysis, then both sets of data are submitted with the original analysis reported.

13.0 CALCULATION

13.1 Response Factor (RF)

$$RF = \frac{As \times Cis}{Ais \times Cs}$$

where:
As = Area of the characteristic ion for the compound being measured.
Ais = Area of the characteristic ion for the specific internal standard.
Cs = Concentration of the compound being measured (ug/l).
Cis = Concentration of the specific internal standard (ug/l).

13.2 Percent Relative Standard Deviation (% RSD)

$$\%RSD = \frac{SD}{RFav} \times 100$$

where:
SD = Standard Deviation
RFav = Average response factor from initial calibration.

13.3 Percent Difference (%D)

$$\%D = \frac{(RFav - RFcv)}{RFav} \times 100$$

where:
RFcv = Response factor from Calibration Verification standard.
RFav = Average response factor from initial calibration.

13.4 Concentration (Conc.)

For water:

$$\text{Conc. } (\mu g/l) = \frac{Ac \times Cis \times Vp}{Ais \times RF \times Vi}$$

For soil/sediment low level (on a dry weight basis):

$$\text{Conc. } (\mu g/kg) = \frac{Ac \times Cis \times Vp}{Ais \times RF \times Ws \times M}$$

For soil/ sediment medium level (on a dry weight basis)

$$\text{Conc. } (\mu g/kg) = \frac{Ac \times Cis \times Vp \times Vt}{Ais \times RF \times Vme \times Ws \times M}$$

Where:
Ac = Area of characteristic ion for compound being measured.
Ais = Area of characteristic ion for internal standard.
Cis = Concentration of internal standard
RF = Response factor of compound being measured(from initial calibration)
Vi = Initial volume of water purged (ml)
Vp = 5 ml (Total Purge Volume)
Vme = Volume of Methanol aliquot
Vt = Ml Solvent + ((100-% solid)/100 x Ws)
Ws = Weight of sample extracted (g).
M = (100 - % moisture in sample) / 100 or % solids / 100

13.5 Percent Recovery (% R)

$$\%\,R = \frac{\text{Concentration found}}{\text{Concentration spiked}} \times 100$$

13.6 Relative Percent Difference (RPD)

$$RPD = \frac{|\,MSC - MSDC\,|}{(1/2)\,(MSC+MSDC)} \times 100$$

Where:
MSC = Matrix Spike Concentration
MSDC = Matrix Spike Duplicate Concentration

13.7 Linear regression by the internal standard technique.

$$C_s = \left(\frac{\frac{A_s}{A_{is}} - b}{A} \right) \times C_{is}$$

Where:
Cs = concentration of target analyte
As = Area of target analyte
Cis = concentration of the internal standard
b = Intercept
a = slope of the line

$$a = \frac{N \Sigma xy - \Sigma x \Sigma y}{N \Sigma x^2 - (\Sigma x)^2}$$

$$b = \frac{\Sigma y - a \Sigma x}{N}$$

N = number of points
x = amount of analyte
y = response of instrument

13.8 Correlation Coefficient

$$r = \frac{\Sigma(x - \bar{x})(y - \bar{y})}{\sqrt{\Sigma(x - \bar{x})^2 \Sigma(y - \bar{y})^2}}$$

Where r = correlation coefficient
x = amount of analyte
y = response of instrument

$\bar{x}$ = average of x values

$\bar{y}$ = average of y values

14.0 DOCUMENTATION

14.1 The Analytical Logbook records the analysis sequence; the logbook must be completed daily. Each instrument will have a separate logbook.

14.1.1 If samples require reanalysis, a brief explanation of the reason must be documented in the Comments section.

14.2 Standards Preparation Logbook must be completed for all standard preparations. All information must be completed; the page must be signed and dated by the appropriate person.

14.2.1 The Accutest lot number must be cross-referenced on the standard vial.

14.3 Instrument Maintenance Logbook must be completed when any type of maintenance is performed on the instrument. Each instrument has a separate log.

14.4 Any corrections to laboratory data must be done using a single line through the error. The initials of the person and date of correction must appear next to the correction.

14.5 Supervisory (or peer) personnel must routinely review (at least once per month) all laboratory logbooks to ensure that information is being recorded properly. Additionally, the maintenance of the logbooks and the accuracy of the recorded information should also be verified during this review.

15.0 POLLUTION PREVENTION & WASTE MANAGEMENT

15.1 Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents, and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids to the environment must be followed. All method users must be familiar with the waste management practices described in section 15.2.

15.2 Waste Management. Individuals performing this method must follow established waste management procedures as described in the waste management SOP, EHS004. This document describes the proper disposal of all waste materials generated during the testing of samples as follows:

15.2.1 Non hazardous aqueous wastes

15.2.2 Hazardous aqueous wastes

15.2.3 Chlorinated organic solvents

15.2.4 Non-chlorinated organic solvents

15.2.5 Hazardous solid wastes

15.2.6 Non-hazardous solid wastes

Table 1: TARGET COMPOUNDS		
Acetone	1,4-Dichlorobenzene	Methylene Bromide
Acetonitrile	Dichlorodifluoromethane	Methylene Chloride
Acrolein	1,1-Dichloroethane	1-Methylnaphthalene (1)
Acrylonitrile	1,2-Dichloroethane	2-Methylnaphthalene (1)
Allyl Chloride	1,1-Dichloroethene	Naphthalene
Benzene	cis-1,2-Dichloroethene	2-Nitropropane (1)
Benzyl chloride	trans-1,2-Dichloroethene	Pentachloroethane
Bromobenzene	1,2-Dichloropropane	Propionitrile
Bromochloromethane	1,3-Dichloropropane	Propyl Acetate (1)
Bromodichloromethane	2,2-Dichloropropane	n-Propylbenzene
Bromoform	1,1-Dichloropropene	Styrene
Bromomethane	cis-1,3-Dichloropropene	Tert Butyl Alcohol
2-Butanone (MEK)	trans-1,3-Dichloropropene	tert-Amyl Methyl Ether
Butyl Acetate (1)	1,4-Dioxane	tert-Butyl Ethyl Ether
n-Butyl Alcohol (1)	Epichlorohydrin (1)	1,1,1,2-Tetrachloroethane
n-Butylbenzene	Ethyl Acetate	1,1,2,2-Tetrachloroethane
sec-Butylbenzene	Ethyl Ether	Tetrachloroethene
tert-Butylbenzene	Ethyl Methacrylate	Tetrahydrofuran
Carbon Disulfide	Ethylbenzene	Toluene
Carbon Tetrachloride	p-Ethyltoluene (1)	trans-1,4-Dichloro-2-Butene
Chlorobenzene	Freon 113	1,2,3-Trichlorobenzene
Chlorodifluoromethane (1)	Heptane (1)	1,2,4-Trichlorobenzene
Chloroethane	Hexachlorobutadine	1,1,1-Trichloroethane
2-Chloroethyl Vinyl Ether	Hexachloroethane	1,1,2-Trichloroethane
Chloroform	Hexane (1)	Trichloroethene
Chloromethane	2-Hexanone	Trichlorofluoromethane
Chloroprene (2-chloro-1,3-butadiene)	Iodomethane (Methy iodide)	1,2,3-Trichloropropane
o-Chlorotoluene	IsoAmyl Alcohol (1)	1,2,4-Trimethylbenzene
p-Chlorotoluene	Isobutyl Alcohol	1,3,5-Trimethylbenzene
Cyclohexane (1)	Isopropyl Acetate (1)	2,2,4 Trimethylpentane
Cyclohexanone	Isopropylbenzene	Vinyl Acetate
di-Isobutylene (1)	p-Isopropyltoluene	Vinyl Chloride
di-Isopropyl Ether	Methacrylonitrile	Vinyltoluene (1)
1,2-Dibromo-3-Chloropropane	Methyl Acetate (1)	m,p-Xylene
Dibromochloromethane	3 Methyl-1-Butanol (1)	o-Xylene
1,2-Dibromoethane	Methyl Tert Butyl Ether	Ethanol
Dibromomethane (1)	Methylcyclohexane	Methyl Acrylate
1,2-Dichlorobenzene	Methyl Methacrylate	1-chloro-1,1-difluoroethane
1,3-Dichlorobenzene	4-Methyl-2-pentanone (MIBK)	1,1,1-trifluoroethane
1,1-dichloro-1-fluroethane	2,2-Dichloropropane	

(1) NELAC Accreditation is not offered for this compound. Results may not be useable for regulatory purposes in States where this accreditation option is not offered.

Table 2. RECOMMENDED OPERATING CONDITION	
Gas Chromatograph/ Mass Spectrometer	
Carrier Gas (linear velocity)	Helium at *30 cm/sec
Mass range	35 – 300 amu
Electron Energy	70 volts (nominal)
Scan time	not to exceed 2 sec. per scan
Injection port temperature	200 - 225 °C
Source temperature	200 - 250 °C
Transfer line temperature	220 - 280 °C
Analyzer temperature	220 - 250 °C
Gas Chromatograph temperature program*	
Initial temperature	*40 °C
Time 1	*3 minutes
Column temperature rate	*8 degrees/min.
Final temperature	*220 °C.- 240 °C
Total run time	*25 – 50 mins
Purge and Trap Device	
Purge time	9 min. (at 40 °C for low-level soil) SIM – 6 min @ 50 °C
Desorb**	4 min. at 190 °C
Bake	>10 min. at 210 °C
Transfer line	100 - 130 °C
Valve temperature	approx. transfer line temperature

* Parameter modification allowed for performance optimization provided operational and QC criteria is achieved.(must be approved by team leader/manager)

** Desorb time may require performance optimum between 2.0 and 4.0 minutes as dictated by trap manufacturers specifications or instrument characteristics

Table 2a - SIM Group Parameters		
Group No.	Retention Time (minutes)	Ions
1	0 – 10.8	58, 65, 66, 88
2	10.8 – 16.0	95, 174, 176, 98, 100, 70

Table 3. BFB KEY IONS AND ION ABUNDANCE CRITERIA	
Mass	Ion Abundance Criteria
50	15-40% of mass 95
75	30-60% of mass 95
95	Base peak, 100% relative abundance
96	5-9% of mass 95
173	< 2% of mass 174
174	> 50% of mass 95
175	5-9% of mass 174
176	>95% and <101% of mass 174
177	5-9% of mass 176

Table 4. INTERNAL STANDARD QUANTITION IONS	
Internal Standard	**Primary/Secondary Ions**
1,4-Difluorobenzene	114 / 63,88
Chlorobenzene-d5	117 / 82, 119
Pentafluorobenzene	168
1,4-Dichlorobenzene-d4	152 / 115, 150
Tert Butyl Alcohol-d9	65/66
Internal Standard (SIM)	
Tert Butyl Alcohol-d9	65/66

Table 5. SURROGATE QUANTITION IONS	
Surrogate Compound	**Primary/Secondary Ions**
1,2 Dichloroethane – d_4	102
Dibromofluoromethane	113
Toluene-d8	98
4-Bromofluorobenzene	95 / 174, 176

Table 6. CRITERIA FOR CCC AND SPCC		
Initial Calibration	Maximum % RSD for CCC is 30 %	
Continuing Calibration	Maximum % D for CCC is 20 %	
Calibration check compounds (CCC)	**Volatile Compound**	
	Vinyl chloride 1,1-Dichloroethene Chloroform 1,2-Dichloropropane Toluene Ethylbenzene	
System Performance Check Compounds (SPCC)	**Compound Name**	**Minimum RF**
	Chloromethane	0.1
	1,1-Dichloroethane	0.1
	Bromoform	0.1
	1,1,2,2-Tetrachloroethane	0.3
	Chlorobenzene	0.3

Table 7. Volatile Internal Standards with Corresponding Analytes Assigned for Quantitation

Analyte	Primary Characteristic Ion	Secondary Characteristic Ion (s)	Analyte	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Tert Butyl Alcohol-d9			Dibromomethane	93	95, 174
Tert Butyl alcohol	59	57	Di-isobutylene	57	
Ethanol	45	46	Epichlorohydrin (pp)	57	57, 49, 62, 51
Pentafluorobenzene			Ethyl methacrylate	69	69, 41, 99, 86, 114
1,1,1-Trichloroethane	97	99, 61	Heptane	57	
1,1-Dichlorethane	63	65, 83	Hexane	56	
1,1-Dichloroethene	96	61, 63	Isopropyl acetate	43	
2,2-Dichloropropane	77	97	Methyl cyclohexane	83	
2-Butanone (pp)	72	43, 72	Methyl methacrylate	69	69, 41, 100, 39
Acetone (pp)	58	43	n-Butanol (pp)	56	41
Acetonitrile (pp)	41	41, 40, 39	Propyl Acetate	43	
Acrolein (pp)	56	55,58	tert Amyl Methyl Ether	73	
Acrylonitrile (pp)	53	52, 51	Toluene	92	91
Allyl Chloride	41		Toluene-d_8	98	
Bromochloromethane	128	49, 130	trans-1,3-Dichloropropene	75	77, 39
Bromomethane	94	96	Trichloroethene	95	97, 130, 132
Carbon disulfide	76	78			
Chlorodifluouromethane	51	86	**Chlorobenzene-d5**	117	82,119
Chloroethane	64	66	1,1,1,2-Tetrachloroethane	131	133, 119
Chloroform	83	85	1,2-Dibromoethane	107	109, 188
Chloromethane	50	52	1,3-Dichloropropane	76	78
Chloroprene	53	53, 88, 90, 51	Bromoform	173	175, 254
cis-1,2-Dichloroethene	96	61, 98	Butyl Acetate	56	
Cyclohexane	84		Chlorobenzene	112	77, 114
Dibromofluoromethane	113		Dibromochloromethane	129	127
Dichlorodifluoromethane	85	87	Ethylbenzene	91	106
Dichloroethane-d_4	102	65	m-Xylene	106	91
Diethyl ether	74	45, 59	o-Xylene	106	91
Diisopropyl ether	45	102	p-Xylene	106	91
Ethyl acetate (pp)	88	43, 45, 61	Styrene	104	78
Ethyl tert Butyl Ether	59		Tetrachloroethene	164	129,131,166
Freon 113	151				
Iodomethane	142	127, 141	**1,4 Dichlorobenzene-d4**	152	115,150
Isobutyl alcohol (pp)	43	43, 41, 42, 74	1,1,2,2-Tetrachloroethane	83	131, 85
Methacrylonitrile (pp)	41	41, 67, 39, 52, 66	1,2,3-Trichlorobenzene	180	182, 145
Methyl Acetate	43	74	1,2,3-Trichloropropane	75	77
Methylene chloride	84	86, 49	1,2,4-Trichlorobenzene	180	182, 145
Methyl-t-butyl ether	73	57	1,2,4-Trimethylbenzene	105	120
Propionitrile (ethyl cyanide)(pp)	54	54, 52, 55, 40	1,2-Dibromo-3-chloropropane(pp)	75	155, 157
Tetrahydrofuran	42		1,2-Dichlorobenzene	146	111,148
trans-1,2-Dichloroethene	96	61, 98	1,3,5-Trimethylbenzene	105	120
Trichlorofluoromethane	151	101, 153	1,3-Dichlorobenzene	146	111, 148
Vinyl acetate	43	86	1,4-Dichlorobenzene	146	111, 148
Vinyl chloride	62	64	2-Chlorotoluene	91	126
Methyl Acrylate	55	85	4-Bromofluorobenzene	95	174, 176
2,2,4 Trimethylpentane	57				
1-chloro-1, 1-difluoroethane	65	45,85			
1,1,1-trifluoroethane	69	69,45			
1,1-dichloro-1-fluroethane	81	45,61			
2,2-Dichloropropane	77	97,79			
1,4 Difluorobenzene	114	63, 88	4-Chlorotoluene	91	126
1,1,2-Trichloroethane	83	97, 85	Benzyl chloride	91	91, 126, 65, 128
1,1-Dichloropropene	75	110, 77	Bromobenzene	156	77, 158

Table 7 Volatile Internal Standards with Corresponding Analytes Assigned for Quantitation

Analyte	Primary Characteristic Ion	Secondary Characteristic Ion (s)	Analyte	Primary Characteristic Ion	Secondary Characteristic Ion (s)
1,2 Dichloroethane	62	98	Cyclohexanone	55	
1,2 Dichloropropane	63	112	Hexachlorobutadiene	225	223, 227
1,4-Dioxane (pp)	88	88, 58, 43, 57	Hexachloroethane (pp)	201	166, 199, 203
2-Chloroethyl-vinylether (pp)	63	65, 106	Isopropylbenzene	105	120
2 – Hexanone	43	58,57,100	Naphthalene	128	-
2-Hexanone (pp)	43	58, 57, 100	n-Butylbenzene	91	92, 134
2-Nitropropane	46	-	n-Propylbenzene	91	120
3 Methyl –1 butanol	55		Pentachloroethane (pp)	167	167,130,132,165,169
4-Methyl-2-pentanone (pp)	100	43, 58, 85	p-isopropyltoluene	119	134,91
Benzene	78	-	sec-Butylbenzene	105	134
Bromodichloromethane	83	85, 127	tert-Buytlbenzene	119	91, 134
Carbon tetrachloride	117	119	trans-1,4-Dichloro-2-butene (pp)	53	88, 75
cis-1,3-Dichloropropene	75	77, 39			
Methylcyclohexane	83		(pp) = Poor Purging Efficiency		

Table 7-1 SIM - Volatile Internal Standards with Corresponding Analytes Assigned for Quantitation

Analyte	Primary Characteristic Ion	Secondary Characteristic Ion (s)
Tert Butyl Alcohol-d9		
1,4-Dioxane	88	58
Toluene –d8	98	100
4-BFB	95	174, 176

Table 8. STANDARDS PREPARATION

A) Internal standard and Surrogate mixtures:

	a) 25/250 µg/ml		b) 250/2,500 µg/ml	
Internal Standard Mixture (2,000 µg/ml)	1.25	ml	1 .25	ml
Tert Butyl Alcohol-d_9 (50,000 µg/ml)	0.5	ml	0.5	ml
Surrogate Mixture (2,500 µg/ml)	1	ml	1	ml
Methanol	97.25	ml	7.25	ml
Total	100	ml	10	ml

- 25/250 µg /ml internal standard and surrogate mixture: The mixture is prepared by measuring 1.25ml of 2,000 µg /ml Internal Standard Mixture (Ultra or equivalent), 0.5 ml of 50,000 µg/ml TBA-d_9 (Absolute or equivalent), 1 ml of 2,500 µg /ml Method 8260A Surrogate Standard Mixture (Ultra or equivalent) and bringing to 100 ml with methanol.

- 250/2,500 µg /ml internal standard and surrogate mixture: The mixture is prepared by measuring 1.25 ml of 2,000 µg /ml Internal Standard Mixture (Ultra or equivalent), 0.5 ml of 50,000 µg/ml TBA-d_9 (Absolute or equivalent), 1 ml of 2,500 µg /ml Method 8260A Surrogate Standard Mixture (Ultra or equivalent) and bringing to 10 ml with methanol.

- 100 µg/ml surrogate mixture: The solution is prepared at 100 µg/ml by measuring 0.4 ml of 2,500 µg/ml Method 8260A Surrogate Standard Mixture (Ultra or equivalent) and bringing to 10 ml with methanol.

- 25/250 µg /ml internal standard mixture: The solution is prepared by measuring 1.25 ml of 2,000 µg /ml Internal Standard Mixture (Ultra or equivalent), 0.5 ml of 50,000 µg/ml TBA-d_9 (Absolute or equivalent), and bringing to 100 ml with methanol.

- 250/2,500 µg /ml internal standard mixture: The solution is prepared by measuring 1.25 ml of 2,000 µg /ml Internal Standard Mixture (Ultra or equivalent), 0.5 ml of 50,000 µg/ml TBA-d_9 (Absolute or equivalent), and bringing to 10 ml with methanol.

B) Bromofluorobenzene (BFB):

	a) 25 µg/ml		b) 250 µg/ml	
BFB (25,000 µg/ml)	0.1	ml	0.1	ml
Methanol	99.9	ml	9.9	ml
Total	100	ml	10	ml

- 25 µg /ml solution for direct injection: The BFB is prepared at 25 µg /ml by measuring 0.1 ml of 25,000 µg /ml (Absolute Stock or equivalent) and diluting to 100 ml with methanol.

- 250 µg /ml solution for purging: The BFB is prepared at 250 µg /ml by measuring 0.1 ml of 25,000 µg /ml (Absolute Stock or equivalent) and diluting to 10 ml with methanol.

Table 8. STANDARD PREPARATION (Continued)

C) Secondary dilution standards:

2nd Dilution Standards	Stock Solution	Concentration (µg/ml)	Volume Added (µl)	Final Volume in Methanol (ml)	Final Concentration (µg/ml)
V8260 Mixture	EPA Method 524.2 Volatiles	2,000	2,500	50	100
	Acrolein	Neat (90%)	66.2		1,000
	Acrylonitrile*	Neat	25		500+
	Propionitrile**	Neat	58.9		1,000++
	Di-iso Butylene	Neat	7.1		100
	Cyclohexane	Neat	6.5		100
	Cyclohexanone	Neat	52.9		1,000
V8260 Custom Mixture	Custom Volatiles Mix A	2,000	2,500	50	100
	Custom Volatiles Mix B	2,000 -100,000	2,500		100 - 5,000
	Epichlorohydrin	Neat	21.4		500
	Iso-Amyl alcohol	Neat	125		2,000
	2-Chloroethyl vinyl ether	Neat	20.1		500
	Ethyl tert-butyl ether	Neat	6.8		100
	Tert-Amyl methyl ether	Neat	6.56		100
	Benzyl chloride	Neat	4.6		100
Gas Mixture	VOC Gas Mixture	2,000	1,000	20	100

- 100 µg /ml V8260 mixture: The mixture is prepared at 100 µg /ml by measuring 2 ml of 2,000 µg /ml EPA Method 524.2 Volatiles stock standard, appropriate amount of some neat compounds, and bringing to 50 ml with methanol.
  * Acrylonitrile = 400 µg /ml (Neat) + 100 µg /ml (EPA Method 524.2 Volatiles)
  ** Propionitrile = 900 µg /ml (Neat) + 100 µg /ml (EPA Method 524.2 Volatiles)

- 100 µg /ml V8260 custom mixture: The mixture is prepared at 100 - 5,000 µg /ml by measuring 2.5ml of 2,000 µg /ml Custom Volatiles Mix A, 2.5 ml of 2,000 - 100,000 µg/ml Custom Volatiles Mix B, appropriate amount of some neat compounds, and bringing to 50 ml with methanol.

- 100 µg /ml gas mixture ***: The mixture is prepared at 100 µg /ml by measuring 1 ml of 2,000 µg /ml stock standard and bring to 20 ml with methanol.
  *** Gas mixture should be prepared weekly.

Table 8. STANDARD PREPARATION (Continued)

D).1 Initial Calibration Standards: using DI water bring to 50 ml final volume: all mixtures used should be **secondary dilution** standards at **100 ppm**.

Standard and Surrogate Concentration	V8260 Mix (100 ppm)	V8260 Custom Mix (100 ppm)	Gas compound Mix (100 ppm)	Surrogate Mix (100ppm)
1 ppb	0.5 µl	0.5 µl	0.5 µl	0.5 µl#
2 ppb *	1.0 µl	1.0 µl	1.0 µl	1.0 µl#
5 ppb	2.5 µl	2.5 µl	2.5 µl	2.5 µl#
10 ppb *	5 µl	5 µl	5 µl	5 µl#
20 ppb	10 µl	10 µl	10 µl	10 µl#
50 ppb	25 µl	25 µl	25 µl	25 µl#
100 ppb	50 µl	50 µl	50 µl	50 µl#
200 ppb	100 µl	100 µl	100 µl	100 µl#
300 ppb *	150 µl	150 µl	150 µl	150 µl#
400 ppb *	200 µl	200 µl	200 µl	200 µl#

* depending upon the instrument.
\# See Section 10.2.2.1 for correction factor.

- When calibrating for Method 5035 low-level soil samples, add 1g of sodium bisulfate to the 40-ml vial before aliquot 5 ml of each standard into vial. This is equivalent to the amount of sodium bisulfate added to the samples and will maintain a consistent purging efficiency of the compounds.

D).2 Initial Calibration Standards for 1,4-Dioxane using SIMS

Standard and Surrogate Concentration (ppb)	1,4-Dioxane Solution (100ppm)	Surrogate Mix (100ppm)	DI Water Final Volume (ml)
2	2 µl	1 µl	100
5	5 µl	2 µl	100
25	25 µl	5 µl	100
50	25 µl	2.5 µl	50
100	50 µl	5 µl	50
200	100 µl	10 µl	50
400	200 µl	20 µl	50

Table 8. STANDARD PREPARATION (Continued)

E) **Continuing Calibration Standard: using DI water bring to 50 ml final volume:** All mixtures used are secondary dilution standards at 100 ppm.

Concentration	V8260 Mix (100 ppm)	V8260 Custom Mix (100 ppm)	Gas compound Mix (100 ppm)
50 ppb	25 µl	25 µl	25 µl

- When calibrating for Method 5035 low-level soil samples, add 1g of sodium bisulfate to the 40-ml vial before aliquot 5 ml of the continuing calibration standard into vial. This is equivalent to the amount of sodium bisulfate added to the samples and will maintain a consistent purging efficiency of the compounds.

F) **Blank Spike (BS): using DI water bring to 50 ml final volume:** All mixtures used are 100 ppm secondary dilution standards.

Concentration	V8260 Mix (100 ppm)	V8260 Custom Mix (100 ppm)	Gas compound Mix (100 ppm)
50 ppb	25 ul	25 ul	25 ul

For lower detection level required (test code: V8260LL)

Concentration	V8260 Mix (100 ppm)	V8260 Custom Mix (100 ppm)	Gas compound Mix (100 ppm)
20 ppb	10 ul	10 ul	10 ul

- When calibrating for Method 5035 low-level soil samples, add 1g of sodium bisulfate to the 40-ml vial before aliquot 5 ml of the blank spike into vial. This is equivalent to the amount of sodium bisulfate added to the samples and will maintain a consistent purging efficiency of the compounds.

Table 9. GUIDELINE FOR DILUTION PREPARATION

Water Sample

Dilution	Sample amount taken	Final volume A (volumetric)	Take from final volume A	Final volume B (volumetric)
1:2	25 ml	50 ml		
1:5	10 ml	50 ml		
1:10	5 ml	50 ml		
1:20	2.5 ml	50 ml		
1: 25	2 ml	50 ml		
1:50	1 ml	50 ml		
1:100	0.5 ml	50 ml		
1:200	250 µl	50 ml		
1:250	200 µl	50 ml		
1:500	100 µl	50 ml		
1:1000	50 µl	50 ml		
1:2000	25 µl	50 ml		
1:2500	20 µl	50 ml		
1:5000	10 µl	50 ml		
1:10000	0.5 ml	50 ml	0.5 ml	50 ml
1:20000	0.5 ml	50 ml	250 µl	50 ml
1:25000	0.5 ml	50 ml	200 µl	50 ml
1:50000	0.5 ml	50 ml	100 µl	50 ml
1:100000	0.5 ml	50 ml	50 µl	50 ml

Soil-Low level (Non-Encore sample)

Dilution	Sample amount taken	Final volume
1:2	2.5 gram	5 ml
1:5	1 gram	5 ml
1:10	0.5 gram	5 ml

Soil-medium level

Additional Dilution	Sample in Methanol amount taken	Final volume (volumetric)
1:1	1 ml	50 ml
1:2	0.5 ml	50 ml
1:5	200 µl	50 ml
1:10	100 µl	50 ml
1:20	50 µl	50 ml
1: 25	40 µl	50 ml
1:50	20 µl	50 ml
1:100	10 µl	50 ml
1:200	5 µl	50 ml
1:250	4 µl	50 ml
1:500	2 µl	50 ml

Table 10. REPORTING LIMITS

Compound	Water µg/l	Soil µg/kg	Compound	Water µg/l	Soil µg/kg
Chlorodifluoromethane	5	5	Chloroform	5	5
Dichlorodifluoromethane	5	5	Freon 113	5	5
Chloromethane	5	5	Methacrylonitrile	10	10
Vinyl chloride	5	5	Butyl Acetate	5	5
Bromomethane	5	5	1,1,1-Trichloroethane	5	5
Chloroethane	5	5	Heptane	5	5
Trichlorofluoromethane	5	5	n-Propyl acetate	5	5
Ethyl ether	5	5	2-Nitropropane	10	10
Acrolein	50	50	Tetrahydrofuran	10	10
1,1-Dichloroethene	2	2	2-Chloroethyl Vinyl Ether	20	20
Tertiary butyl alcohol	50	50	n-Butyl alcohol	250	250
Acetone	5	5	Cyclohexane	5	5
Methyl acetate	5	5	Carbon Tetrachloride	1	1
Allyl chloride	5	5	1,1-Dichloropropene	5	5
Acetonitrile	100	100	Isopropyl Acetate	5	5
Iodomethane	25	25	Benzene	1	1
Iso-butyl alcohol	50	50	1,2-Dichloroethane	2	2
Carbon disulfide	5	5	Trichloroethene	1	1
Methylene chloride	2	2	Methyl methacrylate	10	10
Methyl tert butyl ether	1	1	1,2 Dichloropropane	1	1
Trans-1,2-Dichloroethene	5	5	Di-isobutylene	5	5
Di-isopropyl ether	5	5	Dibromomethane	5	5
2-Butanone	5	5	1,4 Dioxane	125	125
1,1-Dichloroethane	2	2	Bromodichloromethane	1	1
Hexane	5	5	cis-1,3-Dichloropropene	1	1
Chloroprene	5	5	4-Methyl-2-pentanone	5	5
Acrylonitrile	5	5	Toluene	1	1
Vinyl acetate	10	10	trans-1,3-Dichloropropene	1	1
Ethyl acetate	5	5	Ethyl methacrylate	10	10
2,2-Dichloropropane	5	5	1,1,2-Trichloroethane	3	3
Cis-1,2-Dichloroethene	5	5	2-Hexanone	5	5
Bromochloromethane	5	5	Cyclohexanone	5	5

Table 10. REPORTING LIMITS (Continued)

Compound	Water µg/l	Soil µg/kg	Compound	Water µg/l	Soil µg/kg
Tetrachloroethene	1	1	4-Chlorotoluene	5	5
1,3-Dichloropropane	5	5	1,3,5-Trimethylbenzene	5	5
Dibromchloromethane	5	5	tert-Butylbenzene	5	5
1,2-Dibromoethane	2	2	1,2,4 Trimethylbenzene	5	5
Chlorobenzene	2	2	sec-Butylbenzene	5	5
1,1,1,2-Tetrachloroethane	5	5	1,3-Dichlorobenzene	5	5
Ethylbenzene	1	1	p-Isopropyltoluene	5	5
M,p-Xylene	1	1	1,4-Dichlorobenzene	5	5
o-Xylene	1	1	1,2-Dichlorobenzene	5	5
Styrene	5	5	n-Butylbenzene	5	5
Bromoform	4	4	1,2-Dibromo-3-choropropane	10	10
Isopropylbenzene	2	2	1,2,4-Trichlorobenzene	5	5
Bromobenzene	5	5	Hexachlorobutadiene	5	5
1,1,2,2-Tetrachloroethane	2	2	Naphthalene	5	5
Trans-1,4-Dichloro-2-butene	5	5	1,2,3-Trichlorobenzene	5	5
1,2,3-Trichloropropane	5	5	Epichlorohydrin	100	100
n-Proplybenzene	5	5	3-Methyl-1-butanol	5	5
2-Chlorotoluene	5	5	Hexachloroethane	5	5
Ethanol	50	--	Methyl Acrylate	5	--
Benzyl Chloride	1	--	Methylcyclohexane	5	5
2,2,4 Trimethylpentane	5	5			

Table 11. COMPOUNDS THAT MAY EXHIBIT CARRYOVER

Compound
1,2,4-Trichlorobenzene
Hexachlorobutadiene
Naphthalene
1,2,3-Trichlorobenzene

APPENDIX E

AECOM Blank Field Forms

Equipment Calibration/Check Log
Groundwater Development, Monitoring, & Sampling Form

AECOM

Well ID:

Ground Water Sample Collection Record

Client: _____ Date: _____ Time: Start _____ (24hr)
Project No: _____ Finish _____
Site Location: _____
Weather: _____ Collector(s): _____

1. WELL and WATER LEVEL DATA: (measured from Top of Casing)

Total well length: _____ Screen interval: _____ Depth of pump intake: _____
Water table depth: _____ Casing type/diameter: _____ Minimum purge volume: _____ (gals)
Water column length: _____ Tubing length\dia: _____ (calculations on reverse)

2. WELL PURGE DATA

Purge/Sample Method: Bladder pump with dedicated teflon tubing

Acceptance Criteria defined over three consecutive readings (Field Sampling Plan)

- Temperature	3%	- ORP	± 10 mV	- Drawdown	< 0.3 ft
- pH	+/- 1 NTU	- SpCond.	3%		
- D.O.	10%	- Turbidity	<10 NTU or within 10% if >10 NTU		

Field Testing Equipment used:	Make	Model	Serial number(s)
	In-Situ	9500	
	Lamotte	2020	

File name: _____
Begin purge at _____

Time (24hr)	Purge Vol. (Gals)	Temp. (°C)	pH	Spec. Cond. (µS/cm)	ORP (mV)	DO (mg/L)	Turbidity (NTU)	Flow Rate (ml/min)	Drawdown (feet)	Color/odor
	0.00									

(continued on back)

Sample Collector(s): _____ Date: _____

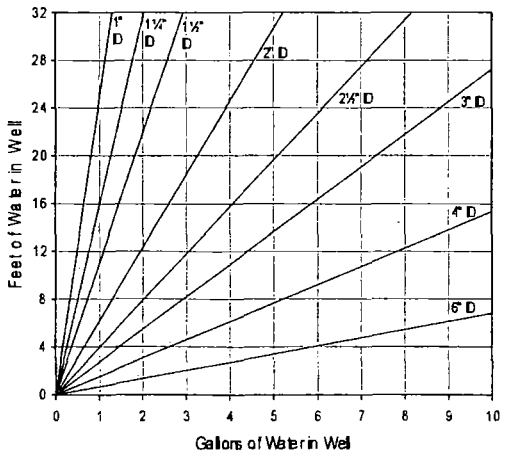
SAMPLE COLLECTION DATA Well ID:

Sample ID	Parameters	Bottles/Preservatives	Time (24 hr)	Flow Rate (ml/min)	Comments
	VOC's	3 - 40mL Clear Vials			

VOCs - Volatile organic compounds

G - Glass
HCl - Hydrchloric acid

PID Well Headspace Readings			
Time (24hr)	Result (ppm)	Time (24hr)	Result (ppm)



Calculations:

tubing length =
x _____ vol. per foot
tubing vol. =
+ _____ (flow cell vol.)
system vol. = [ ] (liters)

x 3 volumes
min. purge vol = [ ] (liters)
x 0.26417 (conversion)
0 gallons

Volume per linear ft of tubing		
ID (in)	Gallon	Liter
0.250	0.0025	0.0097
0.375	0.0057	0.0217
0.500	0.0102	0.0386
0.750	0.0229	0.0869
1.000	0.0408	0.1544
1.250	0.0637	0.2413
1.500	0.0918	0.3475
2.000	0.1632	0.6178
2.500	0.2550	0.9653
3.000	0.3672	1.3900
4.000	0.6528	2.4711
6.000	1.4688	5.5600

Collector(s): _____ Date: _____

Project Name: UTC

Project Number:

Date:_____

AECOM

Calibration Form

Parameter	Instrument	Standard		Stimulus (Std. Value)	Ambient Temp. C	Response (Initial Value)	Final (Adjusted Value)	Initials & Time	Comments
	Manf/Model and SN#	Manf/Model	SN/Exp. Date						
pH 7.00	In-Situ troll 9500 SN	In-Situ Quick Cal Solution #	Lot # Exp. Date	7.00					
				7.00					
				7.00					
				7.00					
				7.00			———		Post Cal (read only profiler mode)
Specific Cond.		In-Situ Quick Cal Solution #	Lot # Exp. Date	7,980 uS/cm					
				7,980 uS/cm					
				7,980 uS/cm					
				7,980 uS/cm					
				7,980 uS/cm			———		Post Cal (read only profiler mode)
ORP		In-Situ Quick Cal Solution #	Lot # Exp. Date						
							———		Post Cal (read only profiler mode)
Neg ORP (Check Std.)		BCl 5 ml Na2S + 500 ml DI H20 (Mix immediately prior to use)	No Serial # Is accurate for only few minutes once prepared (read immediately)	235 mV			———		Check Std. (read only in profiler mode)
				235 mV			———		Check Std. (read only in profiler mode)
				235 mV			———		Check Std. (read only in profiler mode)
				235 mV			———		Check Std. (read only in profiler mode)
				235 mV			———		Check Std. (read only in profiler mode)
Sat'd DO		Oxygen Saturated Tap Water (using the small bubbler prvided by In-Situ							BP= in. Hg
									BP= In. Hg
									BP= in. Hg
									BP= in. Hg
							———		Post Cal (read only) BP= In.Hg
Zero DO		In-Situ Zero DO Solution #	Lot # Exp. Date						BP= In. Hg
									BP= in. Hg
									BP= in. Hg
									BP= in. Hg
							———		Post Cal (read only) BP= in.Hg
Turbidity	LaMotte 2020	LaMotte		0.00 NTU					Post Cal= NTU@
				10.00 NTU					Post Cal= NTU@